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Work Plan

Remedial Assessment Fansteel Metals Muskogee, Oklahoma

Kirkpatrick & Lockhart Pittsburgh, Pennsylvania

Project No. P0111 June 1990





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Earth Sciences Consultants, Inc. One Triangle Drive Export, PA 15632 412/733-3000

3575 Forest Lake Drive Uniontown, OH 44685 216/896-0233

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Work Plan Remedial Assessment Fansteel Metals Muskogee, Oklahoma

#### 1.0 Introduction

This remedial assessment work plan was prepared to assess soil and groundwater quality and to determine the magnitude and extent of potential contaminants of concern at the Fansteel Metals (Fansteel) facility in Muskogee, Oklahoma. Earth Sciences Consultants, Inc. (Earth Sciences) was retained by Kirkpatrick & Lockhart (K&L) to prepare the work plan and conduct the remedial assessment. An understanding of facility history, current environmental conditions, and past operations is necessary to identify potential environmental concerns related to plant operations and to assess remedial action alternatives and their attendant costs. Earth Sciences proposes to apply technically appropriate investigative methods, in conjunction with the utilization of available information regarding plant operations and site conditions, to generate meaningful data on subsurface conditions. The following sections of this work plan present detailed information relative to the site history of the Fansteel facility, the circumstances prefacing this proposed assessment, and the scope of work which will be employed to complete the assessment.

#### 1.1 Site and Process Descriptions

The Fansteel Muskogee plant occupies approximately 110 acres at a location 2.5 miles northeast of Muskogee, Oklahoma (Figure 1). The site lies along the western edge of the Arkansas River (Webber Falls Reservoir) and is bounded on the north by land owned by Muskogee Port Authority, on the south by U.S. Highway 62, and on the west by State Highway 165 and a service road. The facility was developed on alluvial soils and unconsolidated alluvium approximately 20 to 30 feet thick which are underlain by shale bedrock. As expected in an area adjacent to a major river, the water table at the site is shallow. Groundwater flows largely toward the river with minor variations due to topographic influences. Figure 2 presents a site plan of the Muskogee plant prepared from aerial photographs acquired on September 16, 1987.

The climate of the Muskogee area is typical of that found in the eastern section of Oklahoma. The annual rainfall (1951 to 1980 average) is 40.0 inches. A defined wet season occurs each spring with vigorous thunderstorms associated with numerous strong frontal passages. A secondary wet period occurs in September and early October, resulting from a strong flow of moisture-laden air originating over the summer-heated Gulf of Mexico. Frequent severe thunderstorms and the influence of subtropical air masses often cause unusual precipitation producing large quantities of runoff.

Although wind directions vary as weather systems change, they are predominantly southwesterly. Some seasonal patterns are also observed. This section of Oklahoma typically does not experience severe winters. The frost line rarely exceeds one foot and, therefore, temperature extremes usually do not affect production or wastewater treatment processes.

Fansteel's Muskogee plant produced tantalum and columbium metals. Tantalum is used primarily in the electrical/electronics industry in the production of tantalum capacitors. Columbium is marketed for use in heat-resistant alloys. The Fansteel processing facility had been in operation for approximately 30 years until operations ceased in January 1990. The area had not been developed for any use prior to construction of the Fansteel facility and no previous structures existed.

Tantalum- and columbium-bearing ore for processing was procured by Fansteel from several international locations. Additionally, slag containing residual amounts of tantalum and columbium was also acquired from tin smelting operations in Thailand. The drummed ores and slags arrived at the plant via truck and were placed on the facility's barrel storage pad until required for production.

Ore and slag which were digested in the Chemical "C" Building with hydrofluoric acid (HF) underwent a series of leaching, liquid/liquid exchange, and precipitation processes to produce tantalum and columbium oxide. Production processes in the Chemical "A" Building, Chemical "C" Building, and the sodium reduction building employed the following additional reagents: methyl isobutyl ketone (MIBK), sulfuric acid, potassium fluoride, sodium metal, sodium chloride, nitric acid, sodium hydroxide, and ammonia.

Naturally occurring tantalum and columbium ore and the tin smelting slag exhibit low-level radiation from naturally occurring radioactive species contained within them. Accordingly, the Muskogee facility operated under a Nuclear Regulatory Commission (NRC) Source Materials License (License SMB-911) which controls the storage, handling, and processing of ore and slag, the handling of radioactive intermediate products, and the storage of waste products resulting from digestion and extraction of those materials. Other agencies with jurisdiction over facility operations include the U.S. Environmental Protection Agency (USEPA) Region VI, the Oklahoma Water Resources Board (OWRB), and the Oklahoma State Department of Health (OSDH).

Categories of potential environmental concern which have been identified through previous investigations at the Muskogee facility include the following:

- o National Pollutant Discharge Elimination System (NPDES) issues associated with a process water outfall and two storm water outfalls.
- o Low-level radioactive soil contamination from ore and slag storage activities and handling of radioactive materials and wastes at the plant.
- o Acid ponds containing mixed waste (acidic/radioactive) generated by ore and slag processing. A release from Pond No. 3 and residual material which may be present from ponds previously operated in this area may have resulted in releases of hazardous substances to the air, surface water, soils, and groundwater at the facility.
- o Alkaline ponds utilized to store wastes produced by lime treatment of process wastewaters and the associated potential environmental impacts of these structures. Some mixed waste and acidic supernatant generated by ore and slag processing also entered the process wastewater stream for a short period of time.
- o Soil and groundwater contamination from waste product storage units.

Contaminants of concern which have been identified at the site include the following:

o MIBK.

- o Low-level radioactive species.
- o Heavy metals including tantalum, columbium, lead, nickel, antimony, arsenic, barium, cadmium, chromium, mercury, selenium, and silver.
- o Inorganic compounds including hydrogen fluoride, ammonia, calcium fluoride, and sulfate.

The Muskogee plant has been divided into five study areas or source operable units based on process history and probable contaminants of concern to facilitate a logical, scientific, and systematic approach to the investigation. This format, presented in the following sections, allows for a clear and concise description of all work activities proposed for each of the areas and the methods employed to complete them. The study areas outlined in Figure 2 are defined as follows:

- o Study Area I contains the alkaline ponds (Ponds Nos. 6, 7, 8, and 9) with the exception of Pond No. 5.
- o Study Area II contains the Chemical "C" Building, MIBK and acid storage tank farms, the existing acidic pond (Pond No. 3), and the locations of previous acidic ponds.
- o Study Area No. III contains the former ore and slag storage areas. Pond No. 5 is included in this study area because ore storage drums were buried in this area. However, Pond No. 5 also shares similarities with the ponds in Areas I and II.
- o Study Area No. IV is suspected to contain buried storage drums.
- o Study Area No. V contains ammonia storage tanks, barrel and equipment storage, and the bulk of the buildings in which the ore and slag were processed.

Each study area is discussed in detail in Chapter 3.0 of this remedial assessment work plan.

#### 1.2 Project Background

The U.S. Atomic Energy Commission (precursor of NRC) granted Source Material License SMB-911 to Fansteel on January 27, 1967. Fansteel had been operating under this license as amended from that date. The NRC controls discharge of

radionuclides to surface water and storage/management of radioactive materials on site. Discharge of other species is regulated by OWRB under Waste Disposal Permit No. CW-69-020 and by USEPA under NPDES Permit No. OK0001643. OWRB approved a monthly groundwater monitoring plan as part of the waste disposal permit. The Muskogee facility is exempt from regulation under the Resource Conservation and Recovery Act (RCRA) because it is an ore processing facility. However, it is subject to statutory requirements of Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The OSDH and the Occupational Safety and Health Administration also have regulatory authority over certain aspects of facility operations. NRC has primacy over most facility operations; however, OWRB and USEPA may participate in the project if environmental conditions warrant remediation under CERCLA.

On June 18, 1989, a supernatant discharge from Pond No. 3 occurred from the wet well (collection sump) and french drain system adjacent to the subject pond and several seeps near the southwestern corner of Pond No. 3 (Figure 3). The released fluid traveled along the natural drainage course around the western and northern sides of Pond No. 3 and discharged through storm water Outfall 003. Plant personnel immediately mobilized Fansteel employees and local contractors to contain the discharges.

Fluid discharge to the river was terminated by the construction of a temporary dike near Outfall 003 and a second dike near the northwestern corner of Pond No. 3 as shown in Figure 3. Fansteel personnel estimated that approximately 90,000 gallons of fluid were released into the Arkansas River before the discharge was arrested. Fansteel notified the National Response Center, the State Response Commission, Muskogee Local Emergency Committee, and NRC immediately after the release was brought under control and again in writing on June 22, 1989 in accordance with PL99-499 (Superfund Amendments and Reauthorization Act Title III, Section 304) and related regulations. The fluids from the temporary ponds and Pond No. 3 were subsequently removed and routed to the plant's wastewater treatment system as directed by NRC.

A draft outline of a proposed remedial assessment work plan for the Pond No. 3 area entitled, "Remediation Strategy, Pond No. 3" was submitted to NRC, USEPA, and OWRB in March 1990. Preliminary approval of this document was granted by the

regulatory agencies with the stipulation that the entire site be included in the investigation rather than the Pond No. 3 area exclusively. A June 8, 1990 submittal deadline for the remedial assessment work plan to assess conditions throughout the site was requested by the regulatory agencies and approved by Fansteel and Earth Sciences. This document represents the proposed remedial assessment work plan.

#### 1.3 Summary of Previous Investigations

Several investigations have been conducted at the Muskogee site to date including studies to determine the potential environmental effects of the site, NPDES permit applications and related investigations, studies involving closure plans for selected holding ponds, and preliminary investigations involving the release from Pond No. 3. A number of these studies included selected analytical data for soil, waste, surface water, and groundwater sampling points. A description of each pertinent study conducted at the site is outlined below including the purpose of the investigation and a summary of information presented.

A "Preliminary Geology and Hydrology Investigation at the Proposed Location of an Interim Waste Retention Storage Basin" was prepared by Crest Engineering, Inc. of Tulsa, Oklahoma and submitted to NRC in September 1975. This report presented general geologic and hydrologic features of the site for use in the design of Pond No. 5. Several geologic references including structural and stratigraphic, oil and gas, soil, and water resources references were used in the preparation of this report. No conclusions regarding the suitability of the area for use as a settling pond were presented.

A study prepared by Technology Research & Development, Inc. of Oklahoma City, Oklahoma entitled, "Site Hydrology Study" was submitted to Fansteel in May 1983. The purpose of the investigation was to delineate general groundwater flow patterns, determine the hydraulic properties of the alluvial aquifer, to develop a flow model for the site, and to determine if the flow model for the site was impacted significantly by site operations. This information was essential in developing potential contaminant plume pathways associated with any release from the site. The study concluded that the general direction of groundwater movement within the alluvium is towards the Arkansas River parallel to the bedrock

surface, and that the only noticeable alteration of the natural groundwater flow system occurred near Pond No. 3.

Piccolo, Fox and Colby Inc. conducted an "Analysis of Fansteel Inc. Radiological Data" in October 1986. The study presented an analysis of raw data from water and soil samples collected at the site, possible occupational and environmental hazards presented by these findings, and recommendations for monitoring and additional studies. The study concluded that radiation readings in the area were 100 to 1000 times the natural background levels, but that these levels were generally low when compared with other radiation work areas at other sites.

Information obtained during a study conducted by NUS Corporation to determine if the NRC license to operate the tantalum and columbium metallurgical extraction facility would be renewed was presented in a report entitled, "Fansteel Metals, Columbium-Tantalum Facility, Muskogee, Oklahoma, Environmental Information" in June 1986. As part of this license renewal process, Fansteel requested that the amount of source material permitted on site at any one time be increased. This report presented detailed information on plant operations, waste production and treatment, environmental conditions, and possible environmental consequences of the continued operation of the plant. Monthly groundwater and surface water chemistry data from January 1984 through August 1987 were presented from existing site monitoring wells and the three outfalls. Some ammonia, fluoride and low-level radioactive contamination was noted, but the data were highly variable from month to month. The permit renewal application was submitted in 1986 and was still pending in 1989.

A USEPA Field Investigation Team (FIT) visited the site in 1986 to perform a preliminary Hazard Ranking System (HRS) scoring and to perform limited sample collection. Related FIT investigations performed in 1987 and 1988 assessed site conditions in regard to the National Priority List (NPL) eligibility criteria under CERCLA. The results of these studies indicated that the environmental conditions at the Muskogee facility did not meet the criteria for inclusion on the NPL for cleanup under CERCLA at that time.

Radian Corporation of Austin, Texas prepared a "Closure Plan for surface Impoundments at Fansteel Metals" in February 1987 to fulfill reporting

requirements of OWRB. Fansteel had intended to close Ponds Nos. 5, 7, and 8 because they were no longer in service and had been replaced by a larger pond (Pond No. 9). A description of the facility and ponds to be closed; a characterization of the wastes within them; a description of the closure procedures, postclosure monitoring, and maintenance; and a schedule for closure activities were presented in this report. A revised plan was presented in May 1987 which also presented proposed postclosure maintenance and monitoring. This plan has not yet been implemented.

Earth Sciences has conducted several investigations at the site since 1987 including an "Environmental Remediation Investigation Plan, Process and Storm Water Discharges" presented to K&L in December 1987. This report outlined the facility's history, production processes, environmental concerns, and an environmental remediation plan to correct the chemistry of the waters discharging through the three site outfalls to consistently bring them into compliance with the effluent limitations set by the NPDES permit. These limitations had occasionally been exceeded at all three outfalls.

In July 1989, a technical report was prepared by Earth Sciences entitled, "Remediation Strategy, Pond No. 3" in response to the failure of Pond No. 3 and the subsequent contamination of surface soils and supernatant discharge to the Arkansas River. This report presented a summary of the on-site activities prior to, during, and after the release; probable cause for the release; and initial regulatory agency response. Also outlined in the report was a remedial investigation/feasibility study (RI/FS) work scope for this area to determine the overall environmental effects of the release, and a remedial alternative conceptual evaluation and considerations. Cost estimates for the RI/FS were also included in this report.

A geophysical survey of Pond No. 3 was conducted by Earth Sciences personnel in August 1989 to delineate a potential groundwater contaminant plume associated with the June 1989 release from the pond. The study was based on the fact that the spilled wastewater from the pond had a low pH and a high metals concentration, therefore increasing groundwater conductivity values in areas impacted by the spilled wastewater. The extent of the plume was delineated to the north of the pond using data obtained during this study. However,

groundwater impacts to the west, east, and south of the pond could not be determined because of cultural interferences (man-made interference such as buried pipelines or fences). Results of the study were presented in a letter report submitted to Fansteel in August 1989.

Fansteel produced a document in March 1990 entitled, "Environmental Impact Study" which contained process briefs outlining tantalum and columbium processing and sodium reduction procedures used at the Muskogee facility. The study was prepared for the purpose of determining preliminary costs to operate a tantalum and sodium reduction processing plant. It provided a brief overview of each process and the effluent elimination path and constituents, utilizing all existing Fansteel technology and equipment. Process flow diagrams, raw material specifications and usages, and standard yields for finished product were also included.

A review of existing groundwater monitoring data completed by Earth Sciences in the fall of 1989 discovered that monthly groundwater monitoring data exhibited significant variability in both concentration and detection levels of contaminants of concern at locations both downgradient and upgradient of the facility. Sufficient documentation does not exist concerning monitoring well construction, sampling logs, and quality assurance/quality control (QA/QC) procedures implemented during sampling events to adequately explain these variations. Based on this lack of information, the existing monitoring wells at the site cannot be considered to be representative of groundwater quality, nor can concentrations of contaminants at their respective locations be considered accurate. Therefore, the subject wells cannot be used as groundwater sampling points during this investigation. However, historical chemistry data of groundwater collected from these wells do give a general indication of groundwater contaminants of concern and their general plume boundaries at the site. This information has been utilized in the preparation of the laboratory analytical program for the remedial assessment. Selected existing wells will be used to obtain water level measurements to aid in the further delineation of groundwater flow patterns at the site.

#### 1.4 Purpose and Objectives

The proposed remedial assessment will be performed at the Muskogee facility to determine the potential impact of past site operations and existing site conditions on the surrounding environment. The results of this study will be utilized to insure an efficient and environmentally sound closure of the site. Shallow soils, alluvium, bedrock, groundwater, surface water and waste residues will be studied to determine if contaminants of environmental concern exist at the site. Studies will be conducted to determine the hydraulic properties of the alluvial and bedrock aquifers underlying the subject site and to define the horizontal and vertical extent of any potential contaminant plumes identified during the investigation. The concentrations of these contaminants and their associated risks to the environment and human health will be evaluated to determine the necessity of site remediation. Additionally, air monitoring will be conducted during the investigation to evaluate the potential for airborne transportation of contaminants. The goal of the investigation will be to present sufficient data to develop technically feasible and cost-effective remedial alternatives to ensure that any risk to the environment from the identified contaminants of concern will be minimized.

Respectfully submitted,

oseph M. Harrick

Project Manager

Scott C. Blauvelt
Director of Geosciences

JMH/SCB: kmg

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# 2.0 Geology and Hydrogeology

An understanding of the geology and hydrogeology of the site is essential in determining the potential environmental impact of any contaminant release and the applicability and cost of remedial alternatives. This section discusses the geology and hydrogeology of the area as it would pertain to such a release.

# 2.1 Regional Geology

Muskogee County is located on the west plunging faulted nose of the Ozark Uplift, a major tectonic feature extending from eastcentral Missouri to northwest Arkansas and northeast Oklahoma. Many minor folds have been mapped on this geologic feature and block faulting is common east of the Arkansas River. These fault systems are of general geologic interest in the area but are not of concern for the purposes of this investigation.

Geologic units of significance to this investigation may be grouped into two categories: bedrock and alluvium. The term bedrock includes all consolidated sedimentary rocks of Pennsylvanian Age that border or lie beneath the alluvium. The alluvium includes all unconsolidated deposits including terrace and alluvium.

Bedrock is Pennsylvanian Age consisting of mostly thin- to massive-bedded sandstone, shale, siltstone, and limestone. The sandstone beds are hard and well cemented and the shales and siltstones are compact and dense. Units identified in the Muskogee area include the Hartshorne Sandstone, the McCurtain Shale, and the Warner Sandstone in ascending order. Permeability in this type of bedrock is generally low and groundwater movement depends on secondary porosity (joints and fractures) rather than primary porosity (intergranular). Bedrock in the area is nearly entirely overlain by alluvial deposits. The general regional topography of the bedrock beneath the alluvial deposits is relatively uniform with minor variations due to differential erosion. The bedrock outcrop closest to the river is approximately four miles east where the erosional scarp of the Atoka Sandstones and Limestones are exposed.

Terrace deposits having upper surfaces ranging from 20 to 120 feet above the floodplain border the alluvial deposits in segments on both sides of the Arkansas River. These deposits are composed predominantly of silt, fine sand, and small

amounts of coarse sand and gravel near the base. The city of Muskogee is on a terrace segment that extends north and east of the city to the bank of the Arkansas River.

Alluvium is formed in segments along the Arkansas River from 1 to 3 miles wide and 3 to 11 miles long. Deposits of alluvium underlying the floodplain consist of clay, silt, sand, and gravel in proportions that vary locally. A general feature of the alluvium is the gradation in grain size from gravel, or coarsegrained sand near the base of the deposit to silt and clay near the surface. Its total thickness averages 42 feet and its saturated thickness is approximately 25 feet.

#### 2.2 Site Geology

The site is located on the west bank of the Arkansas River which is topographically 50 to 60 feet above the river channel. Geomorphically, this is the cut bank of the Arkansas River at this location. The east side of the river is 40 to 50 feet lower in elevation than the Fansteel site and represents the slip-off slope of the river course at this location.

The Muskogee facility and all the immediately adjacent area is covered by 10 to 30 feet of alluvium as shown in the geologic cross section presented in Figure 4. The cross section was constructed using boring logs, topographic mapping, and other site drawings. It shows approximately 30 feet of unconsolidated alluvial sediments resting on a relatively flatlying Pennsylvanian Age McCurtain Shale surface. The eroded bedrock generally strikes N17°W and dips approximately 3°SW (approximately 200 feet per mile). This dip reflects local structure as the general regional dip is approximately 2°WSW.

Soils at the site are derived from river alluvium, shale, and sandstone. Chateau loam occurs over most of the site on one to three percent slopes. This alluvial soil series consists of deep, dark, moderately well-drained loamy soils with a clayey subsoil, subject to moderate erosion unless protected by vegetation. The Kamic fine sandy loam series also occurs on one to three percent slopes. This is a Savannah range soil found on uplands in the extreme western portion of the site and is deep, light colored, well drained, and moderately sandy with a loamy

Figures 4 and 5. The data used to construct the diagrams were collected in May 1987. Pond No. 3 has since been drained and, therefore, should have little effect on the groundwater table. Although the alluvial aquifer is the predominant water-bearing zone in the area, it is presumed that the shale bedrock is saturated and would produce limited water based on regional hydrogeologic characteristics.

subsoil. Throughout the site, building and impoundment construction has modified the natural soil profile.

# 2.3 Regional Hydrogeology

Shale bedrock permeability is generally low and, therefore, does not readily transmit groundwater in the Muskogee area as discussed previously in Section 2.1 of this work plan. However, a small amount of water is produced from bedrock aquifers throughout the area for domestic and stock use, presumably from fractures or joints within the bedrock. Depths to water measured in wells completed into the bedrock average approximately 30 feet below ground surface.

Alluvium is the most important aquifer in the Muskogee area and along the Arkansas River in general. Precipitation is the primary recharge, averaging approximately 40 inches per year. Natural discharge is mainly by seepage into streams and by evapotranspiration. Quantities of groundwater adequate for domestic or stock use are available almost everywhere on the alluvial floodplain. Wells completed into the alluvium have been recorded to yield as much as 600 gallons per minute.

Groundwater in the alluvium is predominantly a hard, calcium, magnesium bicarbonate type. The quality is affected by precipitation, geology, water movement, and hydraulics of the alluvium. The water is suitable for irrigation and for domestic, stock, and limited industrial purposes.

#### 2.4 Site Hydrogeology

The Muskogee facility was constructed on approximately 10 to 30 feet of alluvium underlain by a dense shale bedrock as shown on the cross section in Figure 4. The alluvium is relatively permeable to groundwater while shale bedrock has a much lower permeability. The water table shown on the cross section indicates that the lower five feet of alluvium are typically saturated with groundwater. Since groundwater cannot readily infiltrate into the underlying shale, it flows along the shale surface and discharges into the Arkansas River where the contact between shale and alluvium is exposed. Shallow groundwater flow directions are generally west to east across the site as shown by the groundwater contour map presented in Figure 5. Effects of the french drain collection system, leakage from Pond No. 3, and possible seepage from Pond No. 5 are also depicted in

# 3.0 Description of Study Areas as Individual Source Operable Units

The Fansteel Muskogee facility was divided into five study areas based upon site operations to allow for a clear discussion of the extent and detail of the work proposed in this remedial assessment. The study areas were described previously in Section 1.1 of this document and are presented in Figure 2. The following sections present detailed physical descriptions and locations of each study area, describe relevant historical facility operations that were conducted within them, and present the specific environmental concerns associated with these historical operations.

#### 3.1 Study Area I - Ponds Nos. 6, 7, 8, and 9

# 3.1.1 Physical Description and Location

Study Area I is the southernmost study area at the site and contains four alkaline process wastewater settling ponds: Ponds Nos. 6, 7, 8, and 9. The settling ponds were placed into service in 1973, 1975, 1978, and 1985, respectively. Ponds Nos. 6 and 7 have clay liners while Ponds Nos. 8 and 9 have single 30-mil synthetic liners and leak detection systems. All of the settling pond's respective dimensions and construction information are listed in Table 1. Pond No. 5 was designed and installed as an alkaline pond also, but was used for various purposes during historical facility operations. Pond No. 5 has accepted treated process wastewater and mixed wastes (radioactive/acidic) typically handled in acidic ponds, and previous information has indicated that storage drums may be buried within its limits. Therefore, it has been grouped in Study Area III because low-level radioactivity has been detected historically in sludge samples collected within its boundary.

### 3.1.2 <u>Description of Past Operations</u>

The surface impoundments in Study Area I were constructed and placed into service as process wastewater settling ponds beginning in 1973. These ponds were constructed to accept treated process wastewater and Pond No. 3 french drain supernatant after lime addition, neutralization, and precipitation. Liquid residue from ore crushing and digestion was routed to Pond No. 3 in Study Area II for retention during normal plant operations. All other waste streams that were collected were routed to the wastewater treatment system, and subsequently

discharged into the alkaline ponds for settling and further pH adjustment. This practice was designed to separate radioactive wastes from nonradioactive wastes.

The treated wastewater was initially routed to Ponds Nos. 8 and 9 for precipitation and then to Ponds Nos. 6 and 7 for additional wastewater clarification. The precipitants utilized in this area were primarily calcium hydroxide and calcium fluoride with the occasional minor addition of several metal hydroxides. After physical separation had occurred, the supernatant was discharged through the NPDES permitted Outfall 001 and the precipitant remained in the ponds. The specific production processes have not changed appreciably since these ponds were placed into service. A flow diagram presenting the sources of water discharged through Outfall 001 is presented in Figure 6.

Based on the knowledge of Fansteel employees in the spring of 1989, the quality of sludge in these ponds could be considered uniform and of known origin. However, when Pond No. 3 failed in June 1989 (as discussed in Section 1.2 of this work plan), the process wastewater stream changed. Fluids from ponds created by the temporary diking of Study Area II and supernatant that remained within Pond No. 3 were routed to the plant's wastewater treatment system as directed by NRC. Following treatment, this material was discharged into Ponds Nos. 8 and 9.

After its failure, Pond No. 3 did not receive the mixed waste stream from ore/slag processing including the semisolid hydrofluoric and sulfuric acid waste residues containing MIBK, heavy metals, and low-level radioactive species (sometimes referred to as "blue mud" as described in Section 3.2.2 of this work plan). Filter presses were put into operation to remove the solid wastes from the acidic process water stream before further processing. Filtered solid wastes were placed in 55-gallon drums and stored on the barrel storage pad in Study Area V. Consequently, neither Pond No. 3 nor any other pond was required for further process use.

#### 3.1.3 <u>Environmental Concerns</u>

Potential groundwater contamination from clay-lined Ponds Nos. 6 and 7 and possible seepage from lined Ponds Nos. 8 and 9 are the environmental concerns posed by Study Area I. Little threat to groundwater is posed by the materials contained within these ponds.

# 3.2 Study Area II - Chemical "C" Process, Ponds Nos, 2 and 3

#### 3.2.1 Physical Description and Location

Study Area II is located in the northern portion of the plant site. Included within this study area are Chemical "C" Building, acidic Pond No. 3 and the related french drain/sump system, former acidic Pond No. 2 and the remains of two former acidic ponds at the same location as Pond No. 3, Outfall 003, and storage tanks for MIBK and HF. Outfall 003 discharges surface water from the subject study area into the Arkansas River. A flow diagram presenting the sources of water discharged through Outfall 003 is presented in Figure 6.

Pond No. 3 has a synthetic liner and the other acidic ponds were lined with clay. It is suspected that some of the residues deposited in the two former acidic ponds may still exist beneath the Pond No. 3 liner as these ponds were not completely removed during Pond No. 3 construction. Physical dimensions and construction details for all settling ponds in existence at the site are presented in Table 1.

# 3.2.2 Description of Past Operations

Pond No. 3 was designed and constructed as a total retention structure for ore/slag residues produced during the digestion and liquid-liquid exchange processes that occurred in Chemical "C" Building. Materials stored in the pond include digested ores and slags and fluid comprised of hydrofluoric and sulfuric acids and containing MIBK, heavy metals, and low-level radioactive species. This waste sludge is sometimes referred to as "blue mud." Former Pond No. 2 and the two former acidic ponds located in the same area as Pond No. 3 accepted the same ore/slag residues which were more recently discharged to Pond No. 3.

Because groundwater was encountered in the alluvium during construction of the pond, a french drain network was installed around the structure to collect groundwater and route it to a sump. A single synthetic liner was installed at the base of the pond with the intent to retain all fluids and residues discharged to the structure. The original design of the french drain collection system allowed groundwater to discharge to the small valley east of Outfall 003. Some time after the pond was placed into service, the pH of the groundwater collected by the french drain decreased, suggesting that Pond No. 3 may have been leaking.

The sump discharge to the local surface water courses was ceased and collected fluids were then pumped from the sump to Pond No. 3 or to the plant's wastewater treatment facility.

The west embankment of Pond No. 3 failed in June 1989, discharging supernatant from the pond into the surrounding area and ultimately into the Arkansas River. The discharge into the river was halted by the emergency construction of containment dikes in the western and northeastern sections of the study area. Residues from this supernatant were deposited over much of the northern and eastern sections of Study Area II.

Chemical processes which occurred in the Chemical "C" Building included the digestion of raw ores and slag and the liquid-liquid extraction process. The processes that were utilized by Fansteel in the Chemical "C" Building are described below. The raw materials containing the tantalum and columbium oxides that were processed by the Fansteel facility consisted of the following types:

- o Tin smelting slag
- o Natural ores
- o Chemically or physically upgraded ores and concentrates

The physical condition and the constituents of the raw materials determine if they require grinding before dissolution. Fansteel had determined that if the raw materials had a silica content of ten percent or greater, the materials did not require milling; this was true of the tin smelting slags. All the natural ores or chemically and physically upgraded ores contain small amounts of silica and therefore required fine grinding to pass through a 200-mesh sieve. The ores were removed from their containers and fed into the ball-type mill by a conveyor belt. The ore was pulverized to the desired mesh fraction by a ball mill and then discharged from the mill through an air classifier. The classifier rejected the coarser materials and returned them by gravity back through a conduit to the feed end of the mill. The finer material was conveyed to a cyclone-type product collector which in turn emptied into metal drums.

The fine ground material was transferred into a feeder hopper with the aid of a barrel dumper. The tared hopper was placed on scales and the prescribed amount of ore was weighed in the hopper. The process flow diagram of ore/slag dissolution through liquid-liquid extraction described below is presented in Figure 7.

The hopper was hoisted into position over the ore or slag feeder for dissolution. The material was fed into a vessel containing HF by an auger-type screw feeder. The dissolution process began when the ore or slag came in contact with 70 percent HF. The contact of the ore or slag with the HF caused an exothermic reaction. This heat governed the length of time it took to feed and dissolve the load of ore or slag. After the addition was complete, the reaction was allowed to cool to 100°F or lower. The slurry was transferred from the digester by a pump through a plate filter press after cooling. The pump lines and the residue in the press were rinsed by pumping water through the lines after the transfer of slurry was complete.

The residue in the press was placed in drums and sampled for tantalum. If the tantalum content was above 1.1 percent in the residue, it was placed in scrap dissolving tanks to remove the remaining tantalum. If the tantalum content was less than 1 percent, it was transferred to the disposal pond (Pond No. 3). Additionally, if the antimony concentration in the liquid was less than ten milligrams per liter, the acidity was adjusted with HF or water and then sent to the feed tanks. If the antimony concentration was greater than ten milligrams per liter, the liquid was required to go through the cementation process to remove the antimony. The only exception to this requirement was if this liquid could be diluted with other feedstock to reduce the concentration of antimony below ten milligrams per liter.

After the tantalum and columbium were separated from the residues and contained in the aqueous solution, the solutions were placed in 10,000-liter rubber-lined steel tanks. These solutions then became feed material for a mixer settler box operation for the separation and purification of the two metals (liquid-liquid extraction). The mixer settler boxes were a series of four boxes with ten chambers with each chamber containing a mixing and settling chamber.

The purpose of the first box was to remove the tantalum and columbium from the aqueous solution with the aid of MIBK and a 15N sulfuric acid solution. All impurities remained in the acidified aqueous solution which was discharged from one end of the box. The MIBK, which had become saturated with tantalum and columbium, was discharged through the opposite end of the box. The aqueous solution was transmitted to another box of the same kind and was stripped of any trace of tantalum and columbium with a clean solution of MIBK. The aqueous solution was discharged to the waste treatment center for neutralization and removal of fluoride.

The tantalum/columbium/MIBK solution was then transferred to another box for separation of the tantalum and columbium metals. A 1.0N solution of sulfuric acid was injected into this box. Through a series of mixing and settling chambers, the columbium was removed from the organic layer and discharged from one end of the box into another box as an aqueous solution. This box removed traces of tantalum by using a clean solution of MIBK. The aqueous solution containing the high-purity columbium was retained in storage tanks to be held until it was ready for further processing. The organic layer containing the tantalum was transferred into another box and mixed with hot deionized water. The tantalum was transferred from the organic solution into the water layer. This tantalum aqueous solution was then contained and held in separate holding tanks until it was ready for further processing. All organic liquids used in the removal of the metals were recycled for use again in the mixer settling box operation.

# 3.2.3 Environmental Concerns

There are three environmental concerns associated with Study Area II. The first area of environmental concern is related to groundwater contamination caused by seepage from Pond No. 3, the pond failure in June 1989, and leachate generated by Pond No. 2 and the former acidic waste ponds. The french drain collection system surrounding Pond No. 3 had intercepted groundwater of degraded quality, suggesting Pond No. 3 had impacted groundwater even before pond failure. Air emissions from Pond No. 3 are a second concern. Such emissions may have contributed to surface water, soil, and groundwater contamination downwind of the pond. A third concern is the impact on surface water on and near the site. Air emissions from Pond No. 3 may be impacting surface water that flows through

Outfall 003, and seepage from former Pond No. 2 and the two former acidic ponds may discharge directly into the Arkansas River.

#### 3.3 Study Area III - Former Storage Areas and Pond No. 5

#### 3.3.1 Physical Description and Location

Study Area No. III is divided into two distinct sections: the former ore and slag storage area and Pond No. 5. The former ore and slag storage area is located in the western section of the site between Study Areas I and IV. Pond No. 5 is a clay-lined settling pond located in the northeastern corner of Study Area I. It has been grouped with the former storage areas for study due to their similar low-level radioactive contamination. However, Pond No. 5 also shares similarities with the alkaline and acidic settling ponds located in Areas I and II, respectively, as it has accepted both types of waste streams.

# 3.3.2 <u>Description of Past Operations</u>

Pond No. 5 is a clay-lined pond originally put into service as an alkaline pond, accepting wastewater streams identical to those discharged to Ponds Nos. 6 through 9. However, in the short interim between the decommissioning of Pond No. 2 and the completion of Pond No. 3, Pond No. 5 accepted acidic wastes. The pond was removed from service in approximately 1975. There is some evidence that storage drums were buried in this pond also. The pond has been designated as a low-level radioactive contaminated area.

The portion of Study Area III west of Study Area V was formerly used to store ore and slag prior to processing. It is suspected that some residual ore and slag may remain on the soil surface. Since the ore and slag have elevated radioactivity, shallow soil may have been contaminated by that residual material.

#### 3.3.3 Environmental Concerns

Low-level radioactive soil contamination presents three environmental concerns. Historically, radioactivity in these areas has been above background and may be above action levels commonly specified by NRC. These conditions restrict land usage, may require remediation, and present a potential hazard to humans by dermal contact and ingestion. The second concern is contamination of surface water and adjoining soils by sediment runoff from affected areas. The third

environmental concern is leaching of radionuclides into site groundwater, possibly causing groundwater contamination. In addition to radioactive leaching, seepage from Pond No. 5 and the drums possibly buried within may contribute contaminants to site groundwater similar to those found in the acidic ponds in Study Area II and the alkaline ponds in Study Area I.

#### 3.4 Study Area IV - Possible Former Drum Storage Area

Study Area IV is located in the western section of the facility adjacent to the northern boundary of the former ore storage area (Study Area III). It is suspected that buried drums of spent ores were disposed in this area. The environmental concern associated with these buried drums is the leaching of materials within them into the subsurface, affecting groundwater and the surrounding soils.

# 3.5 <u>Study Area V - Chemical "A" Process, Wastewater Treatment Plant, Pretreatment Ponds Nos. 1S and 1N</u>

#### 3.5.1 Physical Description and Location

Study Area V is located in the eastcentral section of the site between Study Areas I and II. Chemical "A" Building, the wastewater treatment plant, sodium reduction building, pretreatment Ponds Nos. 1S and 1N, ammonia storage tanks, the barrel storage area, the equipment storage area, and Outfall 002 are all located within the limits of this area. The majority of the chemical processes that were performed at the Muskogee facility, with the exception of ore handling and liquid-liquid extraction, were completed within this area.

#### 3.5.2 <u>Description of Past Operations</u>

Many different processes were accomplished within the limits of Study Area V. Tantalum and columbium production processes incorporating the raffinates resulting from the liquid-liquid extraction performed in Chemical "C" Building, the processing of scrap materials from each of the chemical processes, sodium reduction, and wastewater treatment were all accomplished within their respective buildings within Study Area V. Untreated process wastewater to be routed to the wastewater treatment plant was held in synthetic lined Ponds Nos. 1S and 1N prior to treatment. Barrel storage and equipment storage were also handled within their respective areas. Surface water runoff from the area is discharged to the

Arkansas River through NPDES Outfall 002. A flow diagram presenting the sources of water discharged through Outfall 002 is presented in Figure 6. The following sections present detailed descriptions of the chemical processes that were conducted within this area.

# 3.5.2.1 Scrap Processing

A large amount of scrap was generated and accumulated throughout the tantalum and columbium production process. The scrap was reclaimed by various processes in two different areas at the facility. A flow diagram of the scrap dissolving process is presented in Figure 8.

One facility within this study area handled bulk quantities of scrap such as residues from ore and slag dissolutions, sodium reduction residues, acid powder wash residues, off-specification tantalum powder lots, and columbium press cake. Bulk amounts of this scrap material were processed through 10,000-liter polyethylene-lined steel tanks. HF and nitric acid were used in the processing of this scrap which was subsequently processed by liquid-liquid extraction.

A second facility reprocessed high-purity scrap materials such as bar ends, ingot ends and filings beam melt furnace cleanings, tantalum wire, capacitors, sheet, foil, and other off-specification materials that were purchased from customers that produced tantalum products. Six polyethylene tanks of 60-gallon capacity each were used in dissolving scrap of this type. HF and nitric acid were again used in the scrap dissolving process. Scrap material in one- to two-pound increments was added to and dissolved in the HF until the acid became saturated and the reaction subsided. The solution was allowed to cool and was then transferred to the feed tanks to later be processed through the liquid-liquid extraction process.

#### 3.5.2.2 <u>Columbium Processing</u>

The high-purity columbium solution obtained from the liquid-liquid extraction process was transferred from holding tanks into a 10,000-liter steel rubber-lined tank. Here anhydrous ammonia was sparged into the columbium raffinate to precipitate the columbium. When precipitation was complete, the solution was allowed to cool. The slurry was pumped through a plate and frame press to remove the columbium oxide after cooling. The liquor from the columbium precipitation

was stored in a separate holding tank where any remaining columbium was allowed to settle. The liquor was then removed and routed through a stripping tower for removal of ammonia. Remaining slurry was pumped through a separate plate filter and remaining liquids were transferred to wastewater treatment for removal of ammonia.

The columbium press cake was removed from the press and stored in 55-gallon fiber barrels for drying in a gas-fired calciner. One barrel of this press cake was fed into the calciner on an hourly basis. The exhaust was removed through a water scrubber system to remove any ammonia fluoride before emission to the atmosphere. The dried press cake was then placed in a blender and packaged in 55-gallon fiber barrels lined with plastic and sealed for shipment. A process flow diagram of columbium production described above is presented in Figure 10.

#### 3.5.2.3 Tantalum Processing

The high-purity tantalum solution obtained from the liquid-liquid extraction process was transferred from holding tanks into a 10,000-liter steel polyethylene-lined tank for further processing. A solution of potassium fluoride was added to the high-purity tantalum, precipitating the tantalum to form a potassium heptofluoratantalate ( $K_2TaF_7$ ) crystal. These crystals are permitted to settle and the liquor is syphoned off and placed in a holding tank for further processing. The crystals were centrifuged to remove any remaining liquids and then washed by spraying with a solution of potassium chloride. The liquids removed from the crystals and resulting from the washing was caught and stored in a separate holding tank for further processing. The crystals from the centrifuge are then placed in one of two types of rotary vacuum dryer. One is heated by steam and one by circulating hot oil. After drying, the  $K_2TaF_7$  was placed in 55-gallon fiber drums to be transferred to another area of the facility for further processing through sodium reduction.

All liquors or solutions from the precipitation process were treated with anhydrous ammonia or sodium hydroxide to remove all further traces of tantalum. These solutions are pumped through a plate press to remove or separate crystals from the liquid. The ammoniated water is routed through a stripping tower for removal of ammonia. A process flow diagram of tantalum production described above is presented in Figure 9.

# 3.5.2.4 Sodium Reduction of K<sub>2</sub>TaF<sub>7</sub>

In the late 1950's, the process for controlled sodium reduction of tantalum powder was introduced at Fansteel. This was the first exothermic-type reduction utilizing the addition of sodium to  $K_2TaF_7$  at a controlled rate and temperature. Feed materials for this process were obtained from the drummed  $K_2TaF_7$  production process at the plant. Three types of reductions were made at the Fansteel plant including BV, FM, and TF. A process flow diagram of combined sodium reduction processes is presented in Figure 11.

BV reduction is a high-temperature reduction (965°C) using 1,200.0 pounds of K<sub>2</sub>TaF<sub>7</sub> with 10.0 pounds of tantalum fines, 400 pounds of sodium chloride, and 361.0 pounds of molten sodium with an argon blanket. This was the original type of sodium reduction process used at Fansteel. After the sodium addition is complete, the reduction is cooled and the metal powder cake is crushed, milled, and water washed. The BV milled powder is then transferred to two 500-gallon stainless steel tanks, half full of deionized water. The pH of the wash water is checked and 1.5 liters of hydrochloric acid are added. The tank is then heated to 160°F and agitated for 20 minutes. Ten to 30 minutes are allowed for the powder to settle out in the tank. This process is repeated five times. On the fifth wash, the fluoride concentration of the wash water is checked. Washes continue until the fluoride concentration of the wash water is below 100 parts per million. Once the fluoride concentration has reached the appropriate level and the powder has settled, the wash water is drained from the tank. The washed powder is placed into a vacuum cart to remove all excess water. The powder is then milled and screened and acid washed to eliminate free iron and nickel and to lower the oxygen which accumulated during milling. This powder is produced for use in wire and sheet bar applications. The wastewater from this operation contained fluorides and chlorides. It is drained into a pretreatment holding pond (Pond 1N or 1S) from which it is pumped to the wastewater treatment system.

FM reduction produces a powder used for high charge capacitor applications. This reduction is completed with 1,000.0 pounds of  $K_2TaF_7$ , 800.0 pounds of sodium chloride, 10 pounds of tantalum fines, 100 grams of sodium sulfate, and 298 pounds of molten sodium. The salt is loaded into a nickel-lined retort and dried for eight hours under an argon purge. The temperature is then elevated for one hour and then cooled to 620°C. Sodium is then added at a fast rate to bring the

temperature up to 700°C. A cooling fan is turned on at this time. The rate of sodium addition is then reduced to maintain the temperature until the prescribed amount of sodium is added. The reduction is then cooled, crushed, and milled. The powder is transferred into two wash tanks half full of deionized water. After all the powder has been added, additional deionized water is added to fill the tanks and the agitator is turned on. If the pH of the wash water is greater than 11, 2 liters of hydrochloric acid is added. The tanks are then heated to 160°F. Agitation is stopped after 20 minutes and the powder is permitted to settle. Wash water is drained off and washings are repeated until the fluoride concentration is below 100 parts per million. The washed powder is placed into a vacuum cart to remove excess water, acid washed, dried, and screened. Tests are then conducted on the powder for chemical impurities and capacitance. The wastewater from this operation contains fluorides and chlorides. It is drained into a pretreatment holding pond (Pond 1S or 1N) from which it is pumped to the wastewater treatment system.

The TF reduction is different from the two processes described above in that it utilizes a different type of agitator and salts. Potassium chloride and potassium fluoride are used in 440-pound increments along with 660 pounds of K<sub>2</sub>TaF<sub>7</sub> and 10 pounds of tantalum fines. This material is dried at 300°C with an argon purge for 12 hours. When the material has dried, the temperature is raised and the material is agitated for one hour. The next step is to cool the retort to 800°C and begin the reduction. This reduction required 25 sodium additions of approximately 7.8 pounds at 13-second intervals. After the addition of the prescribed amount of sodium, the material is cooled to room temperature, removed for the retort, crushed, and placed into a large wash tank half full of deionized water with the agitator running. Distilled water and 3 liters of hydrochloric acid are added to fill the tank. The agitation is then stopped and the powder is permitted to settle. When the powder has settled, the water is drained off and four more washings are completed as described above. On the fifth wash, the fluoride concentration of the wash water is checked. Washings continue until the fluoride concentration is less than 10 parts per million. The washed powder is placed into a vacuum cart to remove excess water, acid washed, dried, and The water from the acid wash operations contains fluorides, nitric acid, tantalum powder, and other minute quantities of metal. This effluent is

pressed and the aqueous phase is drained into a pretreatment holding pond (Pond 1S or 1N) from which it is pumped to the wastewater treatment system.

#### 3.5.2.5 Wastewater Treatment

Wastewater produced by the various processes was treated at the wastewater treatment facility east of Chemical "A" Building. Acidic and ammonia wastewaters were held in temporary holding Ponds Nos. 1S and 1N, respectively, prior to treatment. Supernatant was pumped from these holding ponds and treated by employing lime neutralization to remove fluoride by calcium fluoride precipitation. Treated water from this facility was routed to settling Ponds Nos. 8 and 9 in Study Area II.

#### 3.5.3 Environmental Concerns

There are four significant areas of environmental concern within the limits of Study Area V. Potential seepage from the two pretreatment holding ponds could contaminate groundwater and subsurface soils downgradient (toward the Arkansas River) of the ponds. Many open barrels were stored on the barrel storage pad within this area, creating a potential problem with contaminated runoff affecting surface soils. Additionally, spills and overfilling of the sump located in the southwestern corner of Study Area V may also have contributed to surface soil contamination in this area. Historical surface soil chemistry data did identify low-level radioactivity in surface soils near the barrel storage pad and the sump. Radioactivity in these areas may be above action levels commonly specified These conditions restrict land usage, may require remediation, and present a potential hazard to humans by dermal contact and ingestion. The second environmental concern is contamination of surface water and adjoining soils by sediment runoff from affected areas. The third environmental concern is leaching of radionuclides into site groundwater, possibly causing groundwater contamination. A fourth concern is the impact on surface water on and near the Air emissions from the pretreatment ponds and stack/process vapors, contaminated surface soils, and building roof drains may be impacting surface water discharged to the Arkansas River through Outfall 002.

#### 3.6 Background Sampling Locations

Subsurface investigations will be conducted at upgradient locations at the Muskogee facility to establish baseline analytical conditions and to determine

the possible impact of site operations on areas outside the five defined study areas. Background samples will be collected to the west of the site in upwind and hydraulically upgradient areas that had not been used for storage or processing during facility operations. Results of these analyses will be compared to analytical results of samples collected at other locations at the site to determine the impact of Fansteel site operations on the established study areas, if any.

#### 4.0 Description of Scope of Work

Field investigations for the remedial assessment at the Muskogee facility will consist of drilling and soil sampling at 70 locations throughout the site. Of these 70 soil borings, 22 will be completed as shallow wells monitoring the alluvium. Four deep monitoring wells will be completed into the shale bedrock adjacent to four of the shallow monitoring wells. These well "clusters" will aid in evaluating the potential impact of any contaminant plume on groundwater in the bedrock aquifer. Groundwater will be sampled after installation of the monitoring wells. Existing monitoring wells will be not be sampled; eight of the existing wells will be abandoned and the remaining wells will be used for water level measurements to further define the potentiometric surface at the site.

A geophysical survey will be conducted in Study Area No. IV and at Pond No. 5 to determine the locations of drums which may be buried in these areas. Test pits will be excavated and soil will be sampled in Study Area No. IV and possibly in Pond No. 5 at locations determined during the geophysical survey. Other field activities will include sample collection of waste residues at discrete locations in each pond, sediment and surface water sampling at ground surface locations near outfalls and natural surface water discharges, and ambient air sampling before and during field activities to determine the potential impact of the site on air quality. All samples collected will be analyzed for parameters indicative of past site activities by Earth Sciences affiliate laboratory, Antech Ltd. All pertinent field activities will be recorded by Earth Sciences personnel using the standard Field Activity Daily Log presented in Appendix C.

The following sections present descriptions of field activities to be performed by study area and relevant standard field methods to accomplish these tasks. These activities will generate all field data required to complete the initial phase of the investigation, further defining and quantifying the extent of all relevant types of contamination at the site and facilitating the selection of pertinent remedial alternatives. Proposed sample locations are presented in Figure 11. Detailed descriptions of sample collection protocols, decontamination procedures, personal protective equipment, and laboratory methods are presented in the Quality Assurance Project Plan (QAPP) presented in Appendix A.

#### 4.1 Field Methods

The following sections present field methods which will be employed at the Muskogee facility to acquire relevant field data critical to the remedial assessment at the site.

# 4.1.1 <u>Drilling and Soil Sampling</u>

A total of 70 soil borings will be completed at the site to characterize soil conditions at depth. Borings will be advanced using 3-3/4-inch inside diameter hollow-stem augers for borings used to collect soil samples exclusively and 6-1/4-inch inside diameter hollow-stem augers for borings to be completed as monitoring wells. To minimize cross contamination, the drill rig and downhole tools will be steam cleaned prior to the start of drilling activities and between boreholes. Descriptions of subsurface materials encountered during drilling will be noted on standard Earth Sciences boring logs (Appendix C). All drilling activities will be completed under the supervision of a qualified geologist.

All borings will be drilled to auger refusal at the top of shale bedrock. Split-spoon samples will be collected continuously throughout the depth of the bore-hole. Split-spoon sampling equipment will be decontaminated prior to each use to minimize potential sample cross contamination. Soil samples will be screened in the field with a photoionization device (H-Nu) to detect any volatile organic constituents which might be present. Upon completion of soil sampling at soil boring locations where no monitoring well will be installed, the borings will be abandoned by grouting them to ground surface with a cement grout.

Three soil samples will be selected for analysis from each of the shallow borings based on visual inspection, H-Nu readings, and depth sampled. Soil samples designated for laboratory analysis will be transported to the laboratory using standard protocols and analyzed for the list of parameters presented in Section 4.3. Soil samples not selected for laboratory analysis will be archived for future reference.

Permanent eight-inch inside diameter flush-threaded steel casing will be pressure grouted in place from ground surface to the bottom of an approximately two-foot-deep socket drilled into the top of bedrock in the four borings to be completed as deep monitoring wells. Pressure grouting employs the use of a packer inflated

approximately five feet from the bottom of the casing through which cement is pumped under pressure. The packer remains in place during cement curing to provide constant pressure. This procedure provides a reliable seal to prevent mixing of waters from the two aquifers, thereby preventing aquifer cross contamination. After waiting a minimum of 24 hours to permit the cement to harden, the inside of the steel casing will be reamed to remove the cement plug using water rotary techniques. Only potable water from an approved source will be used during drilling operations. The water used for drilling operations will be sampled and analyzed for parameters listed in Section 4.3 to verify that no contaminants of concern have been introduced to the shale bedrock aquifer from this source.

Shale bedrock will be drilled and sampled through the steel casing using core drilling techniques. Drilling will continue to approximately 20 feet below the base of the alluvium. The boring will be reamed to a 7-7/8-inch-diameter using water rotary techniques after coring has been completed. All fluids generated during water rotary and core drilling will be contained and recirculated when possible.

#### 4.1.2 Monitoring Well Installation and Development

A total of 22 shallow monitoring wells and 4 deep "clustered" monitoring wells will be installed at the site. Shallow monitoring wells will be installed in the alluvium at the top of the shale bedrock. Deep monitoring wells will be installed approximately 20 feet below the base of the alluvium in the shale bedrock.

All monitoring wells will be constructed of four-inch-diameter, flush-joint, threaded riser pipe and well screens. Stainless steel well screens and riser pipe will be installed at the background monitoring well location nearest Areas II and V and at all Study Area II and V monitoring well locations. Stainless steel was selected for well construction materials in these study areas due to its relative immunity to MIBK degradation. The topmost section of riser pipe at these well locations will be constructed of Schedule 40 polyvinylchloride (PVC) to simplify the installation of a locking cap. All other monitoring wells will be constructed entirely of PVC well screens and riser pipes. Well screens will be 15 feet in length and factory slotted (0.020 inch) in shallow monitoring wells

and 10 feet in length and factory slotted (0.010 inch) in deep monitoring wells. Each well screen will be fitted with a flush-joint threaded bottom cap constructed of identical material.

After placement of the well screen and riser pipe, the annular space between the monitoring well and the borehole wall will be backfilled with a chemically inert, well-rounded, clean, coarse silica sand sized appropriately for the well screen slot size to a depth of approximately three feet above the top of the well screen. Approximately one to two feet of fine silica sand will be added as filter pack on top of the coarse sand. A layer of bentonite pellets two to three feet thick will be placed on top of the fine sand. Approximately five gallons of potable water from the approved source will then be added to the borehole to hydrate the bentonite pellets if water is not present at this level in the boring. After the bentonite pellets are allowed to hydrate, the remainder of the borehole will be backfilled to ground surface with a cement grout. All monitoring wells will then be completed by installing vented top caps and protective casings with locking caps. Figures 13 and 14 present the proposed shallow and deep monitoring well installation details.

Monitoring wells will be developed using surge-and-bail methods to remove fines and any materials introduced during drilling and well installation. The surge-and-bail method involves the use of a surge block to displace the sand pack followed by bailing two to three well volumes of groundwater to remove the resulting very fine-grained sediment within the well screen. Development will continue until the discharge water is visibly clear of sediment and a sufficient amount of water has been purged to insure that all water introduced during drilling has been removed. After the well reaches acceptable turbidity levels, specific conductance and pH readings will be taken until three consecutive readings vary by less than ten percent. All development water produced will be contained in drums on site.

#### 4.1.3 Monitoring Well Abandonment

As discussed in Section 1.3 of this work plan, based on the lack of information on well installation procedures and past QA/QC procedures, existing monitoring wells at the site will not be utilized to collect groundwater samples during this investigation. Eight of the existing monitoring wells will be abandoned. The

remaining monitoring wells will be used as piezometers only to further delineate the potentiometric surface at the site.

Monitoring wells will be abandoned by first removing the protective casing and trimming the well casing off flush with ground surface. The monitoring well will then be reamed to a 7-7/8-inch diameter using water rotary drilling techniques to the total depth of the well, effectively removing any remaining well construction materials. Water produced during drilling will be contained and recirculated, if possible. The borehole will then be filled with cement grout from total depth to ground surface. A tremie pipe will be used during grouting operations to insure that the cement is accurately placed in the entire length of the borehole. The proposed well abandonment specifications are presented in Figure 15.

# 4.1.4 Geophysical Survey

A geophysical survey using a magnetometer will be conducted in Study Area No. IV and in Pond No. 5 to determine the location of drums that may have been buried in these locations. Magnetometer readings are commonly used to locate buried pipes, drums, tanks, and trenches by measuring changes in the intensity of the earth's magnetic field. The presence of ferrous metals creates variations in the local strength of that field proportional to the mass of the ferrous target.

The intensity of the earth's magnetic field changes daily with sunspots and ionospheric conditions which can cause large and sometimes rapid variations. With time, these variations produce unwanted signals (noise) and can substantially affect magnetic measurements. To diminish the effects of this phenomenon, measurements recorded by a second magnetometer at a fixed location will be utilized to filter out this background noise.

A grid system will be defined in the field to simplify the execution of this study. Typically, a single drum can be detected at distances up to 6 meters while massive piles of drums can be detected at distances up to 20 meters or more. Locations of highest magnetic variation will be selected for test pit excavation to locate and remove any drums which may be buried in these areas.

#### 4.1.5 Test Pit Excavation and Soil Sampling

Test pits will be excavated in Study Area No. IV at locations determined from analysis of geophysical survey data. If magnetic variations exist in Pond No. 5, test pits will be excavated at those locations also. The program of test pit excavations can be implemented concurrently with the drilling program. Test pits will be continuously inspected visually for the presence of buried drums, and care will be taken to minimize damage to any drums that are found while removing them from the excavation.

All test pits will be profiled for depth, subsurface horizons, color (matrix and mottles), structure, moist consistence, rock fragment content, United States Department of Agriculture fine earth textures, and the presence of groundwater inflow to aid in the evaluation of subsurface and groundwater conditions. All excavation activities will be supervised and logged by a qualified soil scientist. Test pits and soil samples collected will be screened with an H-Nu to detect any volatile organic vapors present during excavation. Soil samples will be collected during the excavation for laboratory analysis of parameters presented in Section 4.3 of this work plan. An example of the standard Earth Sciences' test pit log form is presented in Appendix C.

#### 4.1.6 Waste Sampling

The proposed waste sampling plan will consist of collecting waste residue samples from each of the settling ponds at the Muskogee facility. Samples will be collected at discrete intervals beneath the pond surfaces to provide information on the vertical homogeneity of waste residues. Three samples from each of the 17 waste sampling locations presented in Figure 5 (a total of 51 samples) will be collected and analyzed.

A one-inch-diameter Coliwasa sampling device will be used to obtain representative vertical individual-depth waste residue samples. This sampling device is recommended by USEPA in SW-846 for collecting vertical individual-depth and composite samples. The Coliwasa will be lowered into the sludge at the designated locations and pushed to a depth of within one foot of the bottom of the pond or until the vertical resistance is too great to continue manually. This will insure that pond liners are not punctured during sampling. Samples

will be analyzed for parameters of concern listed in Section 4.3 of this work plan.

#### 4.2 Field Activities by Study Area

The following sections present field activities to be completed at the Muskogee facility by study area. All fieldwork will be completed and all samples will be collected in accordance with field methods presented in Section 4.1 and Appendix A of this work plan. Samples will be analyzed for parameters outlined in Section 4.3 of this work plan. Sample types are quantified by study area in Table 2. Study areas and sample locations are presented in Figure 5.

#### 4.2.1 Study Area I

Field activities conducted in Study Area I will include the installation of 7 shallow monitoring wells, 1 deep monitoring well, and 15 soil borings. Stainless steel was selected for well construction materials in this study area due to its relative immunity to MIBK degradation. The topmost section of riser pipe at these well locations will be constructed of Schedule 40 PVC to simplify well construction. A total of 66 soil samples will be selected for analysis from the soil borings and shallow monitoring wells installed in this area (three from each sample location). Soil boring and monitoring well locations were selected to identify and define the possible migration of contaminants from these settling ponds outside of the subject study area.

A total of 12 waste sampling locations have been selected in Ponds Nos. 6, 7, 8, and 9. A total of three samples from each waste sampling location will be selected for laboratory analysis. Five sample locations each have been selected in Ponds Nos. 8 and 9 and one sample location each has been designated in Ponds Nos. 6 and 7 based on the surface area of the ponds. These samples will be analyzed to determine the characteristics of the waste residues deposited within them during past facility operations. A surface water and sediment sample will be collected from Outfall OO1 to determine if the ponds in Study Area I are affecting the current discharge from the outfall and to determine the effects of past operations on the underlying sediment.

#### 4.2.2 Study Area II

The installation of 5 shallow monitoring wells, 1 deep monitoring well, and 13 soil borings will be completed during field activities in Study Area II. Monitoring wells will be constructed of stainless steel well screens and riser pipes. A total of 54 soil samples will be selected for analysis from the soil borings and shallow monitoring wells installed in this area (three from each sample location). Two soil boring locations were designated in the former Pond No. 2 area to characterize waste residues and soils remaining in the area after closure. Four soil borings were located in the downgradient area affected by the June 1989 release from Pond No. 3. The remaining soil boring locations and all Study Area II monitoring well locations were selected to identify possible migration outside of the study area of contaminants relating to Pond No. 3, former Pond No. 2, and the remains of the two settling ponds that were partially removed during Pond No. 3 construction.

Two waste sampling locations have been selected within the limits of Pond No. 3. Three samples from each location will be analyzed to determine the characteristics of the waste residues deposited within them during past facility operations. A surface water and sediment sample will be collected from Outfall 003 to determine the effects of the remaining on-site residues on the surface water discharging through the outfall and the underlying sediment.

#### 4.2.3 Study Area III

A total of 18 soil samples will be collected for laboratory analysis from 6 soil borings installed during field activities in Study Area III. Designated soil boring locations were evenly distributed throughout the study area to provide the most comprehensive coverage possible. Analysis will be performed to determine the possible impact of ore storage in the study area on the underlying soils.

Three sludge samples will be collected and analyzed from the designated sample location in Pond No. 5 to determine the characteristics of the remaining waste residues within the pond. A geophysical survey will be conducted within the limits of the pond to determine the presence of any buried drums. Any sites identified as suspected buried drum locations during the geophysical survey will be excavated in accordance with standard test-pitting procedures. All drums found during the excavations will be carefully removed from the area and stored

at a field designated on-site hazardous materials storage area until the contents can be determined.

#### 4.2.4 Study Area IV

A geophysical survey will be conducted within the limits of Study Area IV during initial field activities in this area to identify the possible locations of buried storage drums. Approximately 13 test pits will be excavated within the study area incorporating these locations. All drums found during the excavations (if any) will be carefully removed from the area and stored at a field designated on-site hazardous materials storage area until the contents can be identified. Method of disposal of any drums excavated will be determined based on waste characterization analyses. One soil sample from each of the test pits will be submitted for laboratory analysis.

# 4.2.5 Study Area V

Field activities conducted in Study Area V will include the installation of 5 shallow monitoring wells, 1 deep monitoring well, and 14 soil borings. Stainless steel was selected for well construction materials for the two westernmost shallow monitoring wells in this study area due to its relative immunity to MIBK degradation. The topmost section of riser pipe at these well locations will be constructed of Schedule 40 PVC to simplify well construction. All other monitoring wells in this area will be constructed of Schedule 40 PVC well screens and riser pipes.

A total of 57 soil samples will be selected for analysis from the soil borings and shallow monitoring wells installed in this area (three from each sample location). Soil boring and monitoring well locations were selected to identify the impact of the barrel storage area, the sump, Chemical "A" Building processes, and Ponds Nos. 1S and 1N on soil and groundwater and to identify possible migration of contaminants relating to these site features and processes.

Three waste samples each will be collected for laboratory analysis from Ponds Nos. 1S and 1N. These samples will be analyzed to characterize the remaining waste residues deposited within their boundaries during past facility operations. A surface water and sediment sample will be collected from Outfall 002 to determine if the ponds in Study Area I are affecting the current discharge from

the outfall and to determine the effects of the remaining on-site residues on the surface water discharging through the outfall and the underlying sediment.

#### 4.2.6 Background Locations

Several soil, groundwater, surface water and sediment samples will be collected at upgradient locations at the Muskogee facility to establish baseline analytical conditions and to determine the possible impact of site operations on areas outside the five defined study areas. Five shallow monitoring wells and one deep "clustered" monitoring well will be installed during this phase of the investigation. Three upgradient sediment and surface water samples will be collected west of the site at locations of natural surface water discharges. A reconnaissance of the river bank will be performed to locate any groundwater seeps that may be present. If located, water from these seeps will be sampled. Results of these analyses will be compared to analytical results of samples collected at other locations at the site to determine the impact, if any, of facility operations on the established study areas.

# 4.2.7 <u>Comprehensive Site Activities</u>

The following sections describe field activities which will take place throughout the site regardless of study area.

#### 4.2.7.1 Groundwater Sampling

Groundwater sampling will be performed at all monitoring well locations after well development is complete in accordance with the sampling and analysis plan presented in Appendix A. Representative samples will be collected from both the alluvial and shallow bedrock aquifers. A comparison of groundwater chemistry data from each aquifer at each well cluster will be performed to determine if vertical migration of groundwater contaminants has occurred. An example of the standard Earth Sciences Well Evacuation/Water Sampling Report, the Analytical Work Request Forms, and Chain of Custody forms is included in Appendix C.

#### 4.2.7.2 Hydrogeologic Testing

Hydrogeologic testing will be conducted to determine representative hydraulic properties of both the shallow alluvial and shale bedrock aquifers at the Muskogee facility. This information is valuable in defining possible contaminant pathways, determining the potential environmental risk associated with

groundwater contamination, and in developing technically feasible remedial alternatives for groundwater remediation. Methods which incorporate appropriate hydraulic testing without significant discharge of contaminated groundwater have been selected.

Hydraulic conductivity, storativity, specific yield, transmissivity, maximum and minimum hydraulic gradients, and average linear flow velocity will be calculated for both the alluvial and shale bedrock aquifers beneath the site. Hydraulic conductivities will be calculated using aquifer type (confined or unconfined) and hydraulic test (slug or pump) specific models. Storativity, specific yield, and transmissivity for each aquifer will be calculated using standard formulas and aquifer characteristics determined during drilling activities. A potentiometric surface map will be generated for each aquifer from data collected during the remedial assessment. The maximum and minimum hydraulic gradients will be determined using information provided on these potentiometric surface maps. To determine the rate of groundwater migration beneath the site, the average linear flow velocity in the downgradient direction will be calculated using the formula:

$$V = \frac{k * i}{n_o}$$

where

V = average linear flow velocity,

k = hydraulic conductivity,

i = groundwater flow gradient, and

 $n_e$  = effective porosity.

#### 4.2.7.2.1 Slug Tests

Slug tests will be performed on 19 of the newly installed monitoring wells at the site after development. Fifteen of the shallow wells and the four deep wells will be slug tested to characterize the hydraulic properties of both the alluvial and shale bedrock aquifers. The tests will be performed by placing a solid PVC pipe (slug) below the static water level and measuring the subsequent rate of fall of the water level in the well. In-Situ Hermit digital environmental data loggers interfaced with pressure transducers will be used to record the rate of

water level recovery in the monitoring wells during the testing periods. It is suspected that groundwater in both the alluvial and shale bedrock aquifers are under unconfined conditions at the site. Therefore, recovery data generated during these tests will be reduced using the H. Bouwer and R. C. Rice method (1976, "A Slug Test for Determining Hydraulic Conductivity of Unconfined Aquifers with Completely or Partially Penetrating Wells," Water Resources Research, Vol. 12, No. 3) to determine the hydraulic conductivity (K) of the aquifers. Appropriate computer modeling software will be used to aid in these calculations. A copy of the standard Earth Sciences Slug Test Data form is included in Appendix C.

# 4.2.7.2.2 Pumping Tests

Pumping tests will be conducted in a background location determined in the field by Earth Sciences personnel. Ideally, this location will not have been impacted by site operations in either the alluvial or shale bedrock aquifers. The pump tests will require the installation of four two-inch inside diameter PVC observation wells and one four-inch-diameter PVC pumping well in the shale bedrock aquifer and three two-inch-diameter PVC observation wells and one four-inch-diameter PVC pumping well in the alluvial aquifer. Only an initial decontamination of drilling equipment and no soil sampling activities will be necessary because the proposed pumping tests will be conducted on wells at background locations. Pumping wells will be drilled and installed utilizing the standard procedures presented in Sections 4.1.1 and 4.1.2 of this work plan. All drilling and pumping activities will be supervised by a qualified hydrogeologist.

Individual pumping tests will be performed for each aquifer. Pumping test procedures will be identical for both the alluvial and shale bedrock aquifers, assuming that the shale bedrock aquifer can produce a well yield of at least five gallons per minute. After well development following procedures outlined in Section 4.1.2 of this work plan, an electric submersible pump will be lowered into the well and a step test will be conducted to determine maximum well yield. Upon completion of the step test, the water level in the pumping well will be allowed to equilibrate and a steady rate nonequilibrium pumping test will be performed in the applicable aquifer. The pumping well will be pumped at approximately 75 percent of the maximum well yield. Observation wells will be positioned to enable completion of the pumping test within 48 hours of the start of

continuous pumping, if possible. Depending on aquifer conditions, these tests could require as much as 120 hours to complete. Water level measurements from the pumping and observation wells will be measured and recorded using In-Situ Hermit digital environmental data loggers interfaced with pressure transducers during the pumping test and throughout the water level recovery period following completion of the pumping test. Data will be reduced using Jacob's Straight-Line Method and other appropriate methods to determine the hydraulic properties of each aquifer. Applicable computer modeling software will be used to aid in these calculations.

#### 4.2.7.3 Air Monitoring

High volume samplers will be utilized to determine quantities of airborne emissions from the site. Five background monitoring locations will be established at or near the site; one upwind and four downwind of the site. Monitoring will be conducted continuously for approximately one week prior to the start of remedial assessment activities. Additional air sampling will be conducted for one week during the active phase of the investigation at locations downwind of Study Areas Nos. I, II, and V. Sampling locations downwind of the site will be close to site operations due to the general westerly wind direction and the proximity of Webber Falls Reservoir (Arkansas River).

Gravimetric analysis of particulates retained by the air filters will be conducted by a local qualified laboratory. If no statistically significant difference between upwind and downwind air sampling locations is noted, no further analysis will be performed. Particulate analyses for fluoride, gross alpha, gross beta, columbium, tantalum, lead, nickel, arsenic, cadmium, and chromium will be performed in the event that gravimetric testing indicates a statistical difference between upwind and downwind locations.

#### 4.2.7.4 Monitoring Well and Sample Location Survey

A licensed surveyor will be subcontracted to survey all groundwater, surface water, soil, sediment, and waste sampling locations at the Muskogee facility. The horizontal and vertical position of each location will be referenced to United States Geologic Survey (USGS) coordinates. Elevation measurements tied to a USGS datum and referenced to mean sea level will be taken at the top of well casing and at ground surface at each monitoring well location. The survey point

on the well casing will be permanently marked and surveyed to the nearest hundredth of a foot at each monitoring well to insure consistent water level data.

#### 4.3 <u>Laboratory Analysis</u>

Samples will be collected and transported for analysis at the Muskogee facility following standard procedures outlined in Appendices A and B. Contaminants of concern at the site have been defined based on past site operations and historical groundwater, soil, and waste chemistry data. All samples collected will be analyzed by Earth Sciences' affiliate laboratory, Antech, Ltd.

Groundwater, seep, and surface water discharge samples collected for laboratory analysis will be analyzed for dissolved metals (tantalum, columbium, lead, nickel, antimony, arsenic, barium, cadmium, calcium, chromium, mercury, selenium, and silver), total fluoride, total ammonia, total sulfate, gross alpha, gross beta, and MIBK. Additionally, 30 percent of the groundwater samples collected will be analyzed for the USEPA Target Compound List (TCL) parameters to verify that the contaminants-of-concern list identified at the site is comprehensive.

Soil, sediment, and waste samples collected for laboratory analysis at the site will be analyzed for total metals (tantalum, columbium, lead, nickel, antimony, arsenic, barium, cadmium, calcium, chromium, mercury, selenium, and silver), total fluoride, total ammonia, total sulfate, gross alpha, gross beta, and MIBK. Additionally, 20 percent of these samples will be analyzed for the USEPA Toxicity Characteristic Leachate Procedure metals (TCLP) to determine the mobility of any contaminant detected.

#### 5.0 Quality Assurance/Quality Control

In addition to the analysis of duplicate samples, matrix spike, matrix spike duplicates, trip blanks, decontamination blanks, and field blanks to monitor the validity of accuracy of the analytical data, the following sample handling procedures will be performed to insure that the integrity of the samples is maintained. Detailed descriptions of sample collection techniques, decontamination procedures, and the quality assurance project plan are presented in Appendix A. Copies of the well evacuation/water sampling report, Chain of Custody Record, and Laboratory Analyses Request forms are included in Appendix C.

#### 5.1 Sample Labels

Each label will record the sample number, location, and depth; the time and date of collection; and the sampler's initials.

#### 5.2 Field Log Books

A field log book will be maintained to provide an accurate record of all samples collected. At a minimum, the following information will be recorded:

- o Date and time
- o Sampler's name
- o Sample identification and type
- o Sample point location
- o Sampling equipment and sample recovery
- o Sample description
- o Miscellaneous comments (stain, odor, etc.)

#### 5.3 Sample Shuttles

The laboratory will provide sample shuttles for on-site sample storage and shipment to the laboratory. Individual sample vials/jars will be packaged with vermiculite to prevent breakage and to insure sample stability during transit. Shuttles will be cooled to approximately 4°C using "Blue Ice" or ice sealed in leakproof containers. Chain of Custody and Sample Analysis Request forms will be sealed in a waterproof bag and sealed in the shuttles.

#### 5.4 Chain of Custody

Chain of Custody Record forms will be filled out with indelible ink for each sample collected. The forms will accompany the samples as they change

possession. The original form will be sealed in a waterproof bag and enclosed in the sample shuttle. A copy of the form will be retained by Earth Sciences. The field sampler will insure that possession or sight of samples is maintained until they are dispatched via overnight (24-hour) express mail to the laboratory.

# 5.5 Chain of Custody Seals

Sample shuttles will be securely closed and chain of custody seals placed over the lid so that contents cannot be accessed without breaking the seal. The sampler will record his signature and the date and time on each seal in indelible ink.

#### 6.0 Remedial Assessment Report Outline

Upon the completion of all field and analytical work, Earth Sciences will prepare a final report documenting the results of the remedial assessments and providing conclusions relative to the magnitude and extent of identified contamination. Additionally, recommendations will be presented outlining future management activities including additional investigations and/or appropriate and applicable remediation activities (if necessary). Following is an outline of the anticipated contents of the subject report:

- o Executive summary
- o Introduction
- o Scope of work and field activities
- o Geologic and hydrogeologic setting
- o Occurrence and distribution of contaminants
- o Data validation summary
- o Conclusion.
- o Recommendations

# 7.0 Project Schedule

A bar graph illustrating the tentative project schedule is presented in Figure 16. As shown on the graph, one week will be required for project mobilization (field office setup, subcontractor notification, materials acquisition, and shipping) prior to the initiation of field activities. It is expected that all fieldwork specified in the remedial assessment work plan including drilling soil borings; installing and developing monitoring wells; geophysical surveying; test pit excavating; and soil, groundwater, surface water, waste, and sediment sampling will take nine weeks to complete. Laboratory analysis of samples collected at the site will commence approximately one week after fieldwork initiation and continue for three weeks after fieldwork completion. Approximately five weeks will be required to complete the remedial assessment report after the receipt of all laboratory data.

**Figures** 

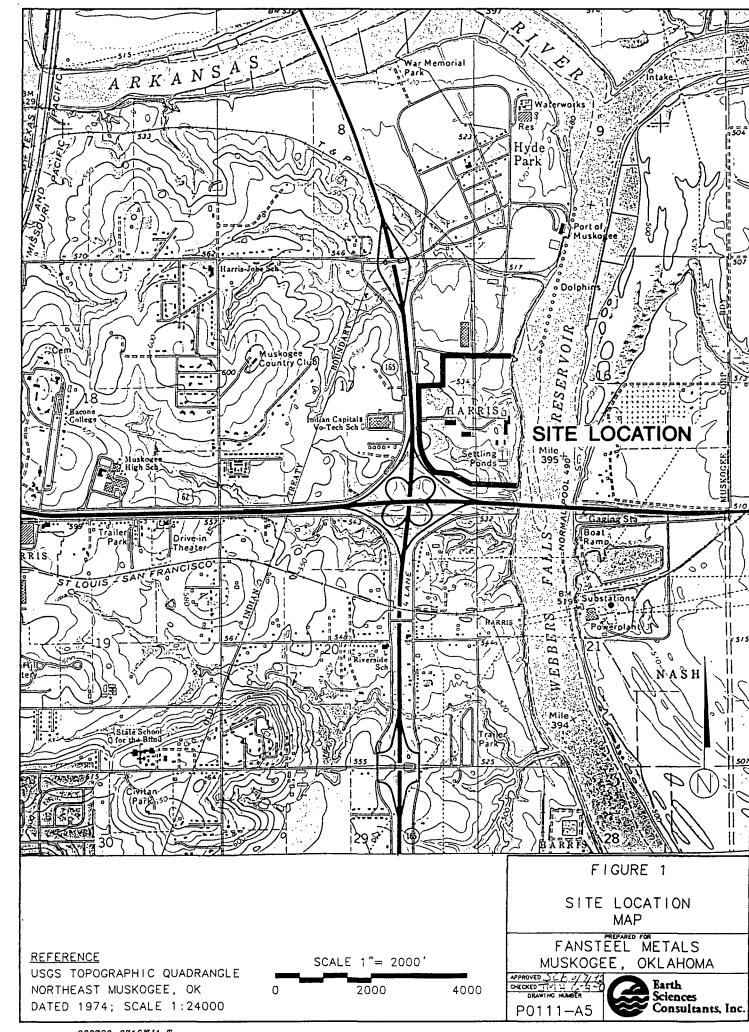
Table 2 Sampling Summary Remedial Assessment Muskogee, Oklahoma

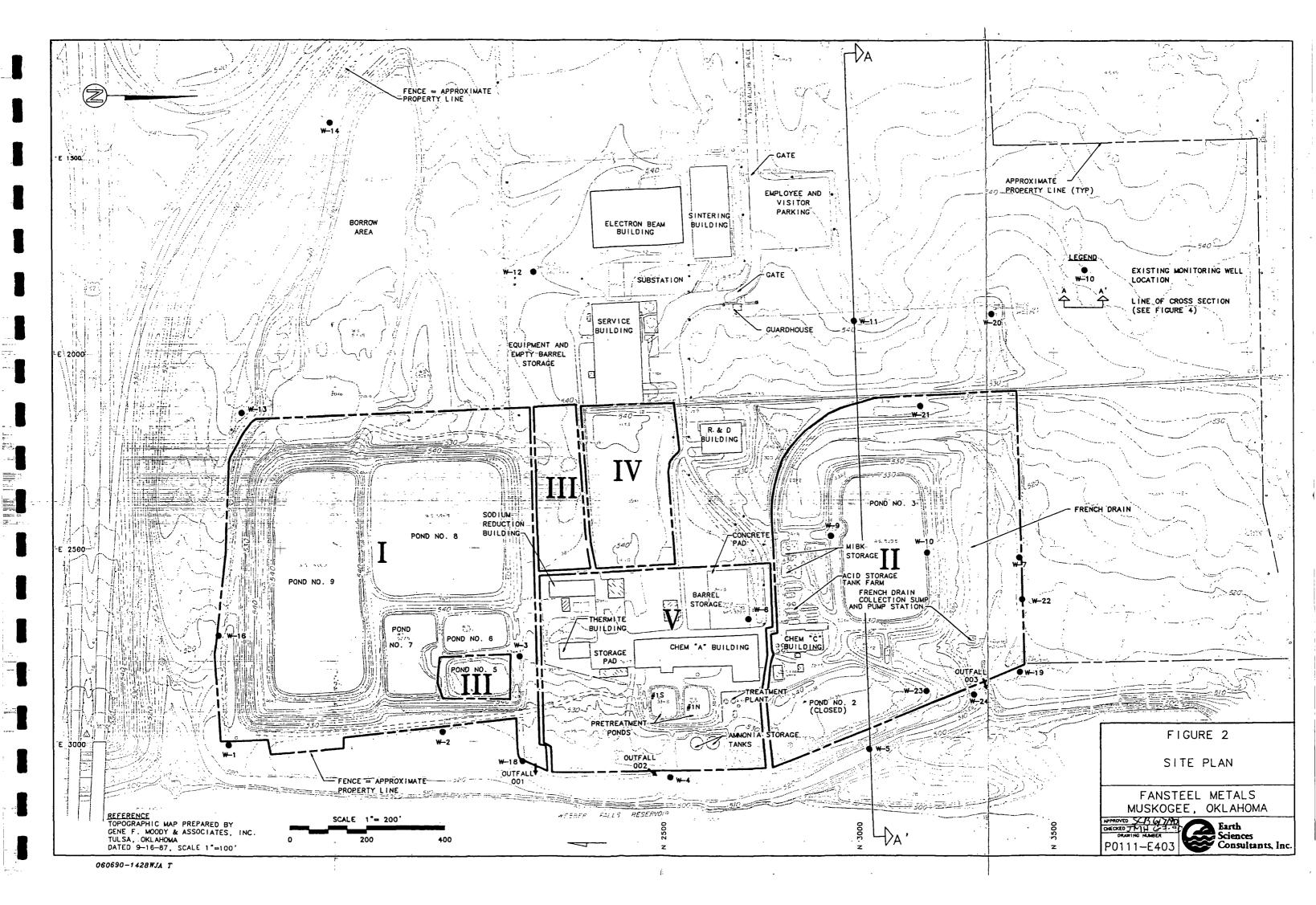
Sample Types	Study Areas						
	Area I	Area II	Area III	Area IV	Area V	Background	
Shallow Monitoring Wells	7	5	_	-	5	5	
Deep Monitoring Wells	1	1	-	-	1	1	
Waste Sample Locations	12	8	1	-	2	-	
Soil Borings	15	13	6	-	14	-	
Test Pits	-	-	-	13	-	-	
Sediment Samples	1	1	-	-	1	3	

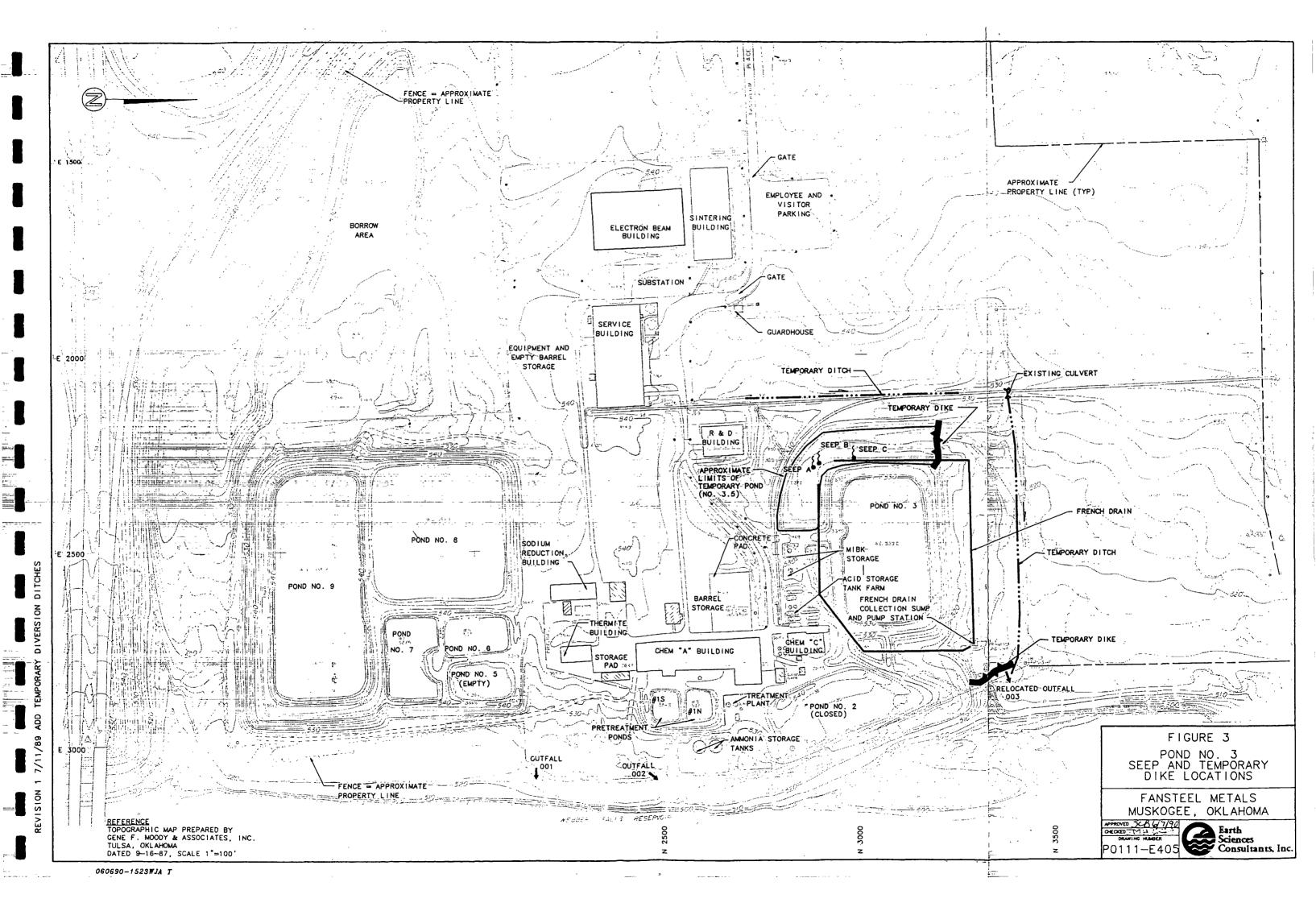
Table 1
Dimensions and Construction of Settling Ponds
Muskogee, Oklahoma

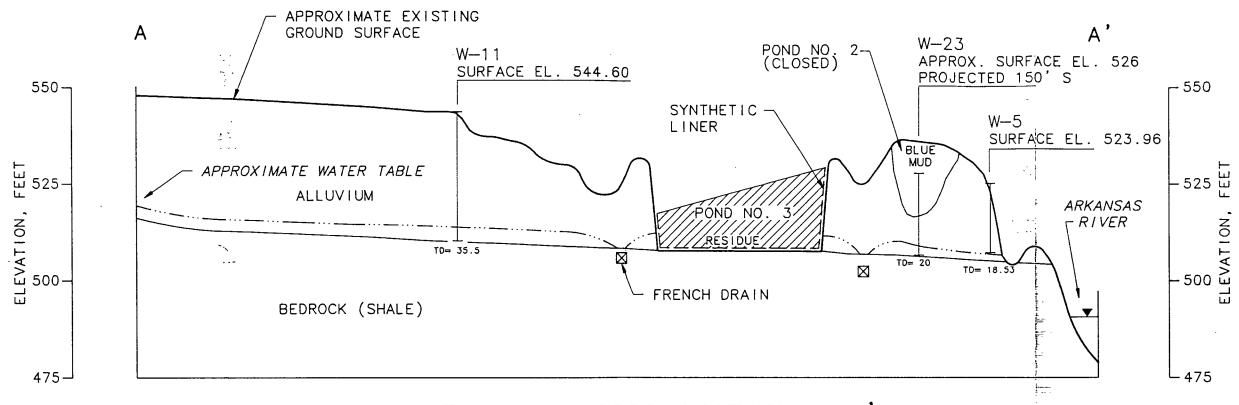
	Dimensions		Approximate	·		Approximate Year
Pond No.	Length (ft)	Width (ft)	Depth (ft)	Liner Type	Leak Detection	Placed into Service
110.	(20)			Brief Type	Deceerion	<u> </u>
1S	90	80	10	Synthetic	No	1981
1N	80	80	10	Synthetic	No	1981
2	350	150	25	Clay, capped with one PVC sheet, one polyethylene sheet, and 6 to 12 inches of soil	No	1960
3	400	250	25	Synthetic	No	1979
5	200	100	9	Clay	No	1973
6	200	100	9	Clay	No	1973
7	250	150	7	Clay	No	1975
8	350	350	25	Synthetic	Yes	1978
9	600	250	20	Synthetic	Yes	1985

Tables







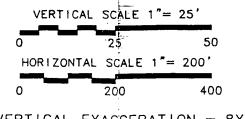


GEOLOGIC CROSS SECTION A-A' (LOOKING NORTH)

THIS CROSS SECTION DEPICTS GEOLOGIC CONDITIONS AT SPECIFIC LOCATIONS SHOWN BASED UPON AVAILABLE DATA. GEOLOGIC CONDITIONS AT OTHER LOCATIONS MAY VARY.

# **NOTES**

- 1. FOR PLAN LOCATION OF CROSS SECTION, SEE FIGURE 2.
- 2. ELEVATIONS SHOWN ARE BASED ON EXISTING SURVEY DATA AND TOPOGRAPHIC MAPPING PREPARED BY GENE F. MOODY & ASSOCIATES, INC. DATED 09-16-87, SCALE 1"= 100'.
- 3. RESIDUE THICKNESS IN POND NO. 3 IS ESTIMATED.



VERTICAL EXAGGERATION = 8X

FIGURE 4 GEOLOGIC CROSS SECTION A-A'

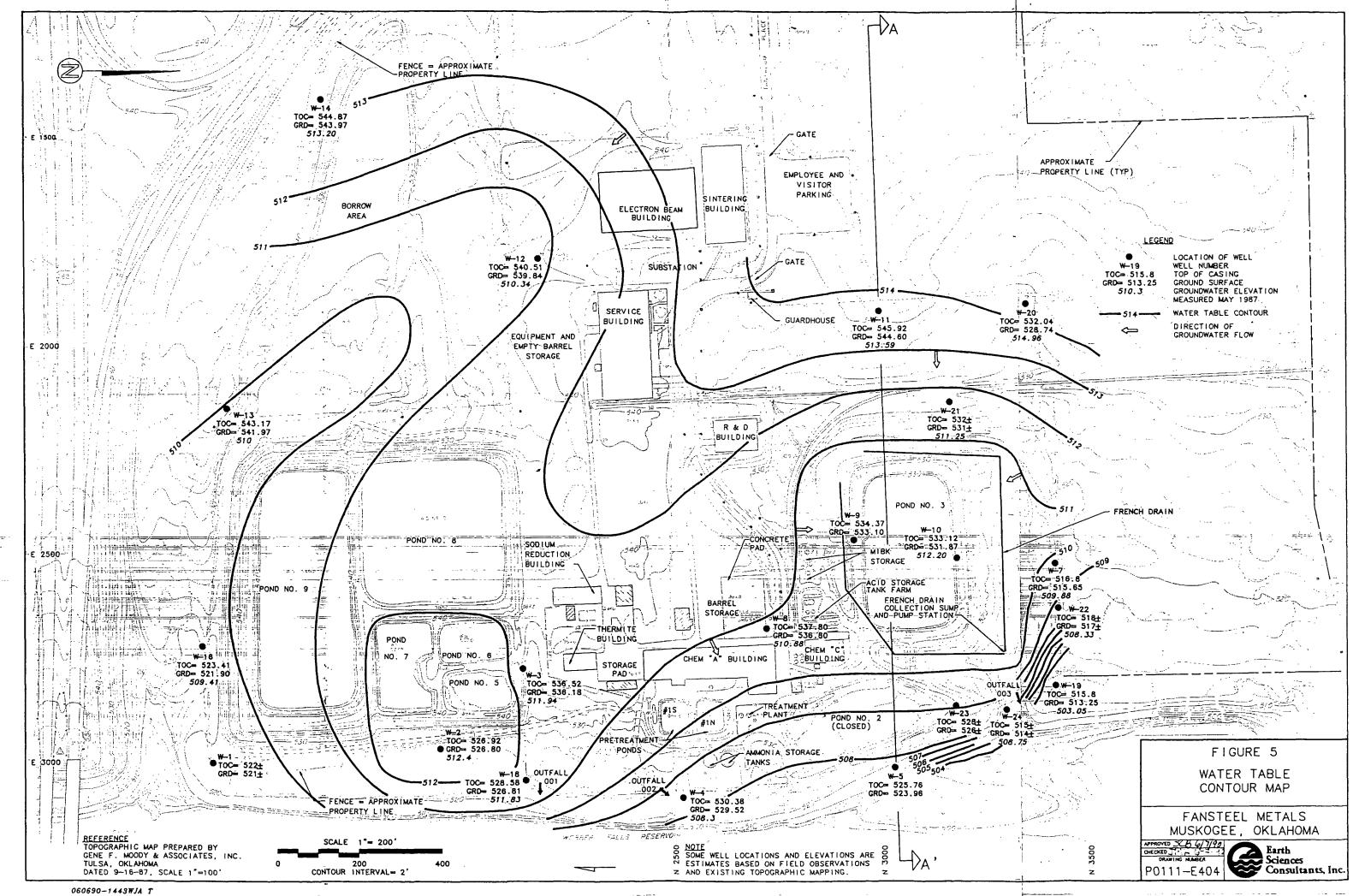
FANSTEEL METALS MUSKOGEE, OKLAHOMA

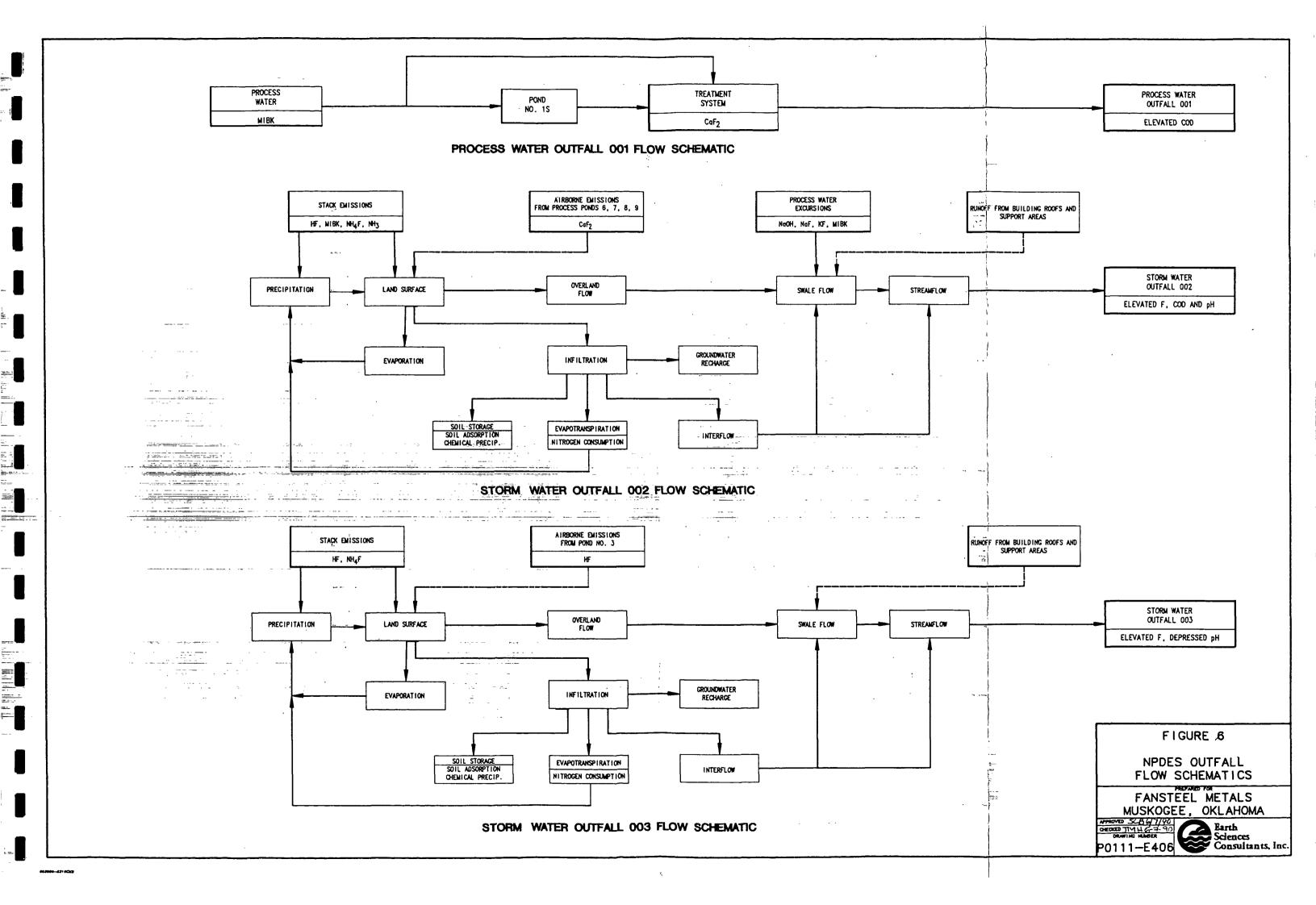
APPROVED CHANGE Barth

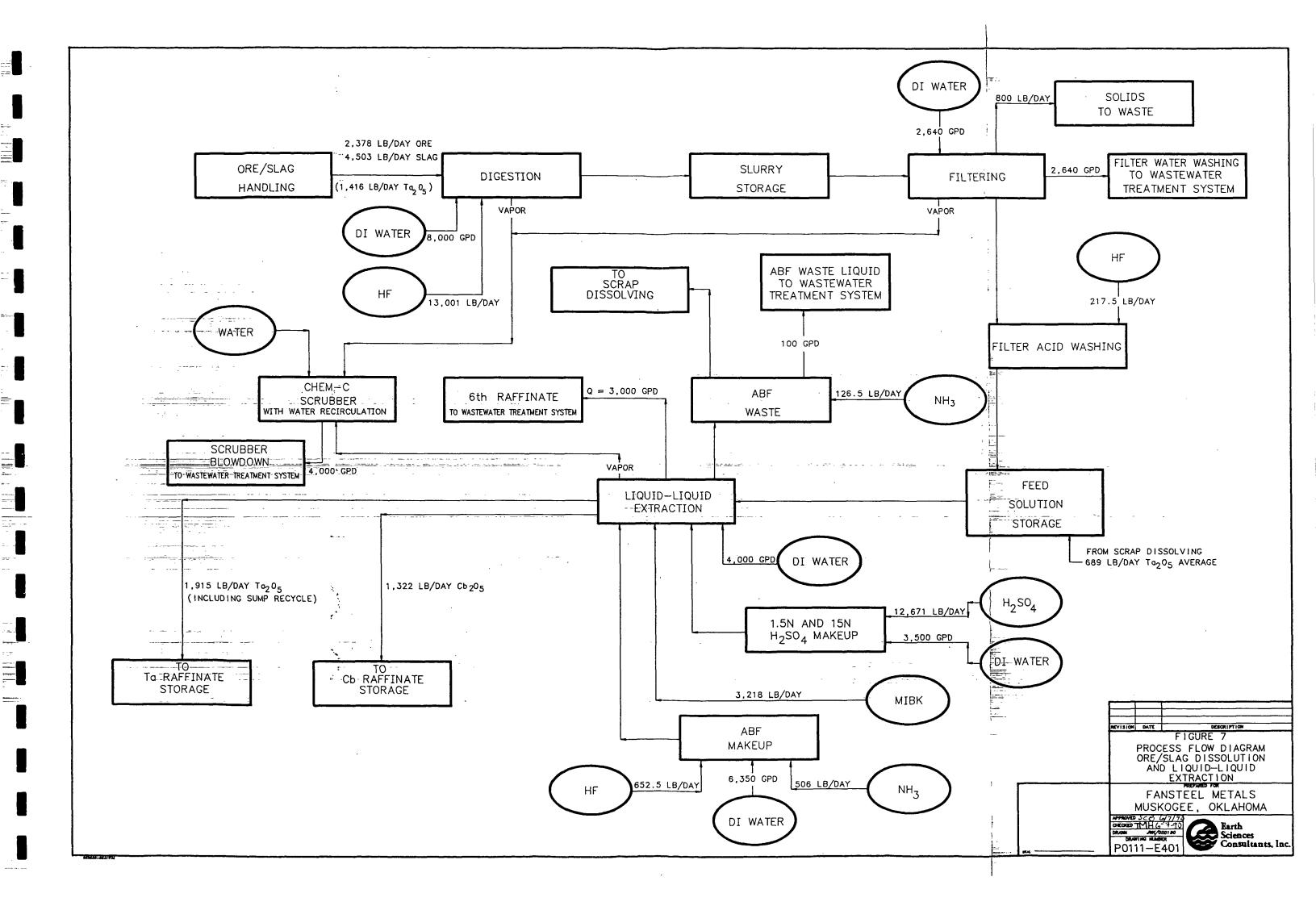
ORIGINAL MARGER

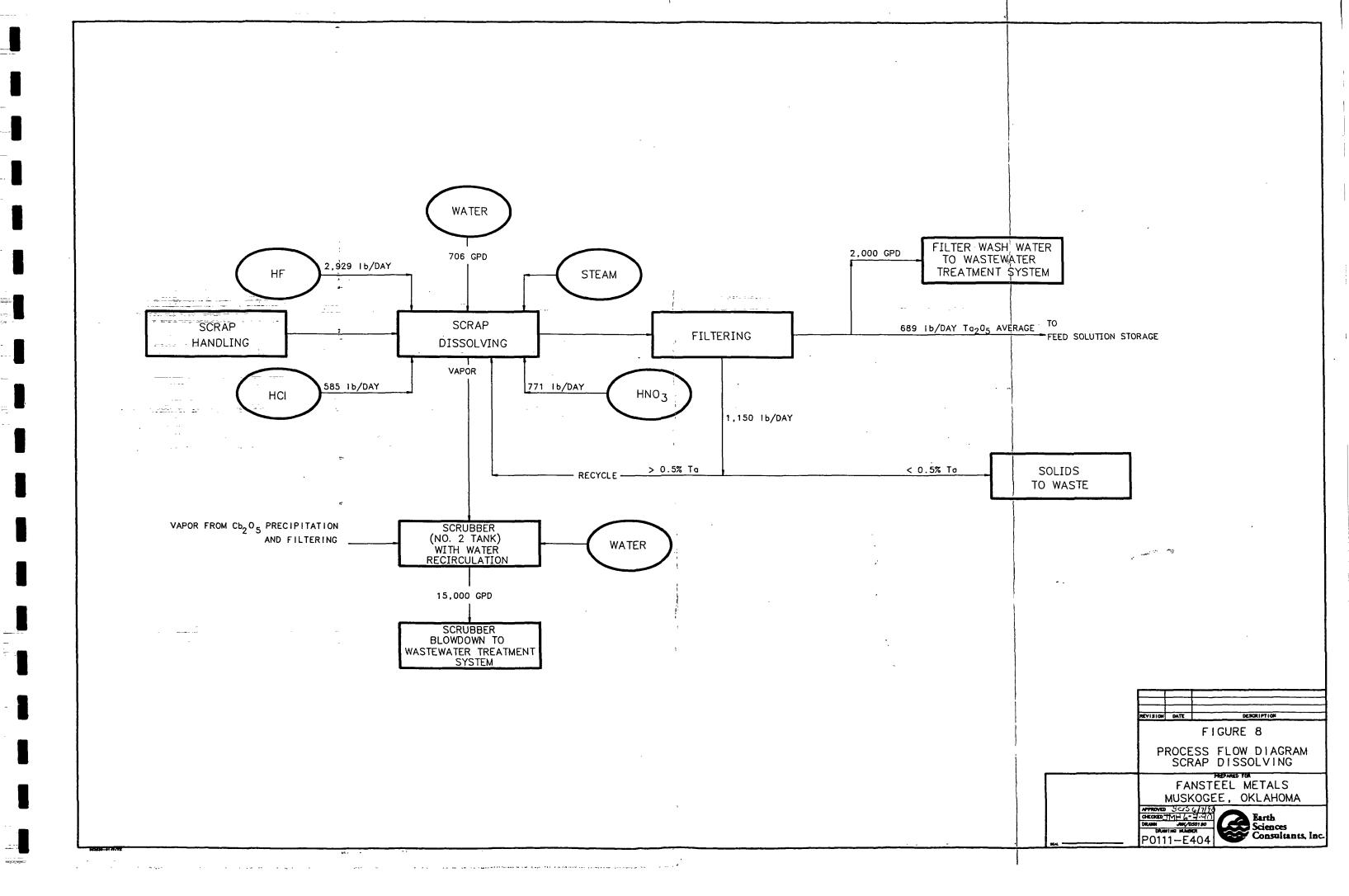
DRAWING MARGER

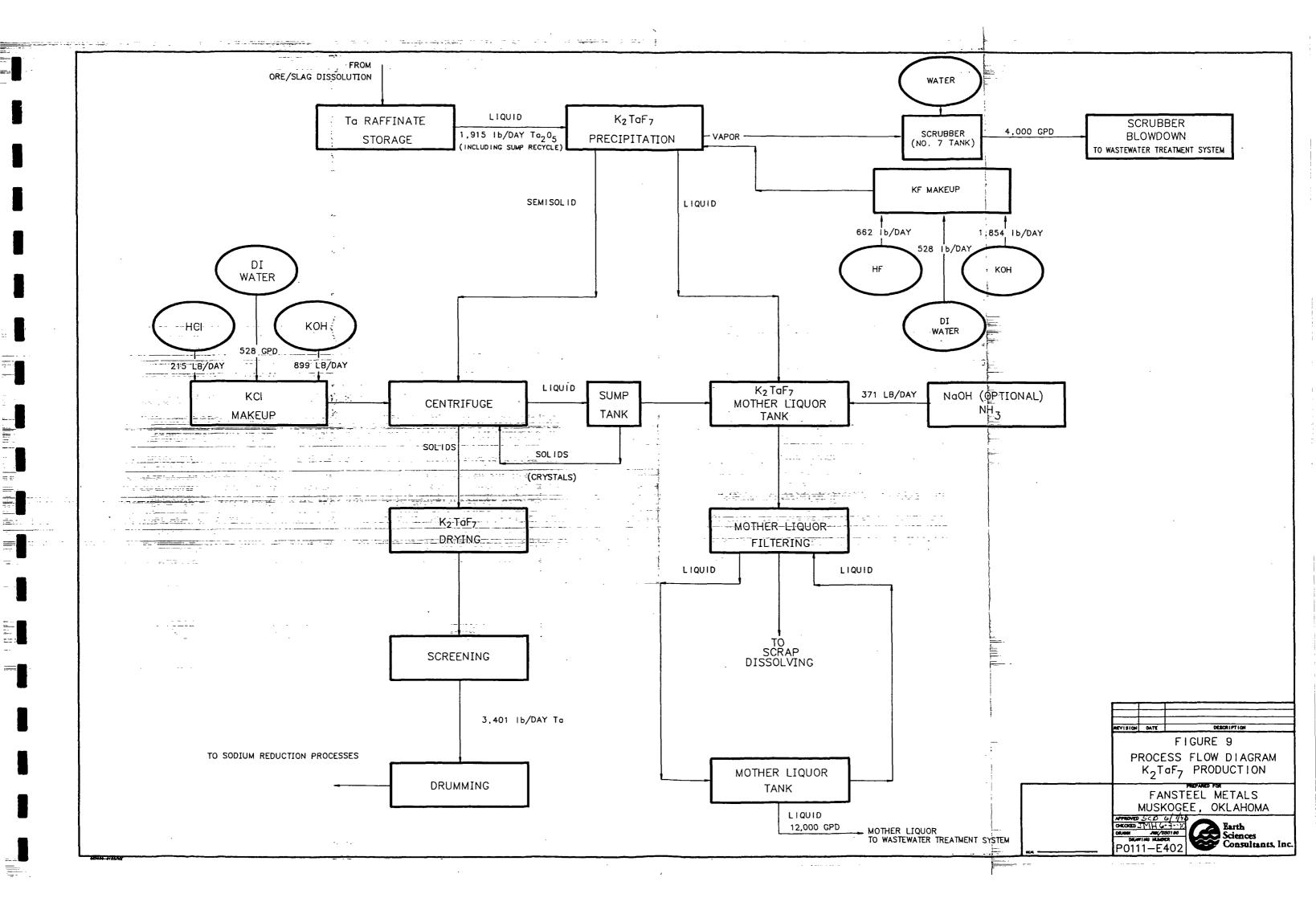
Consultants, Inc.

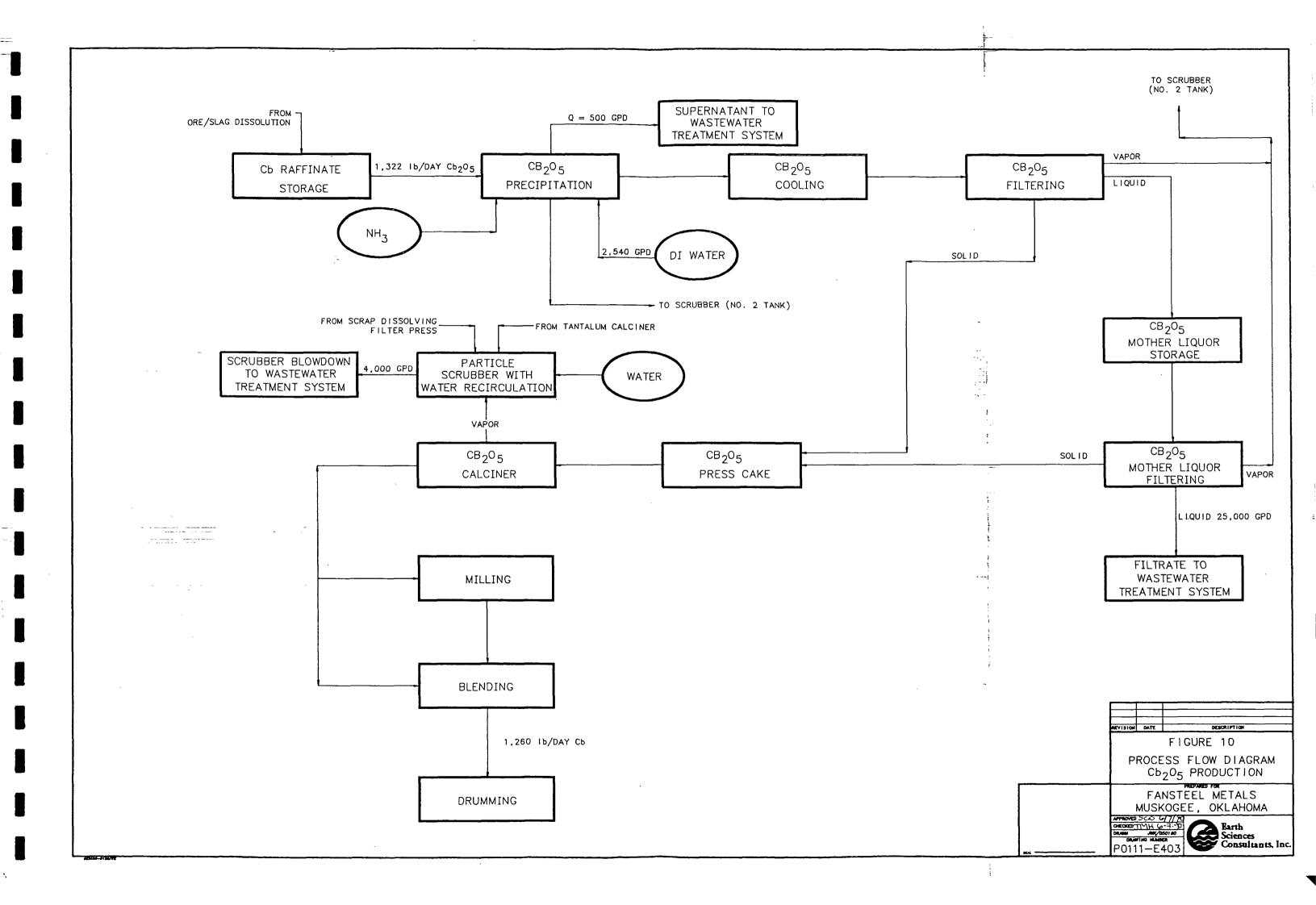


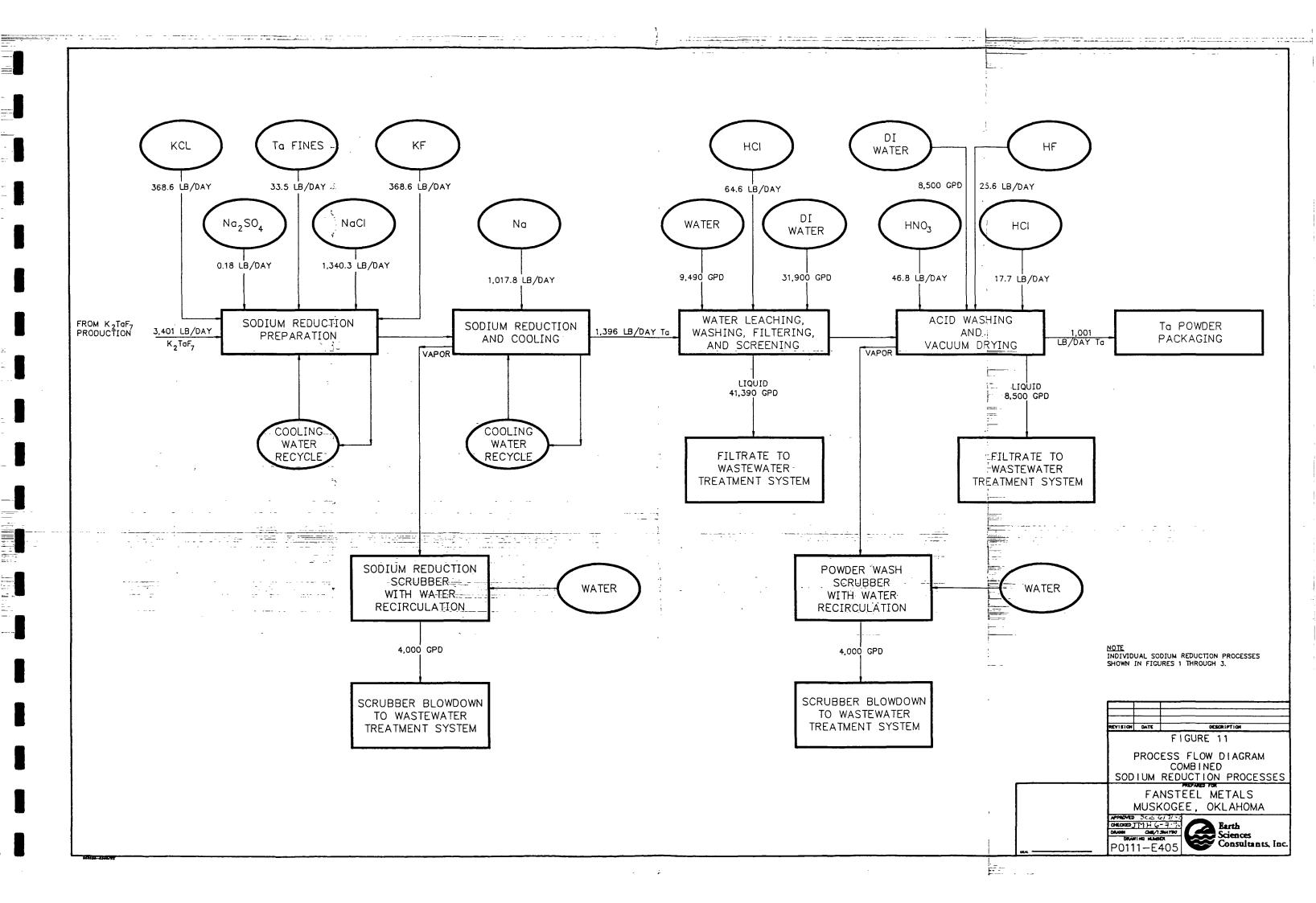


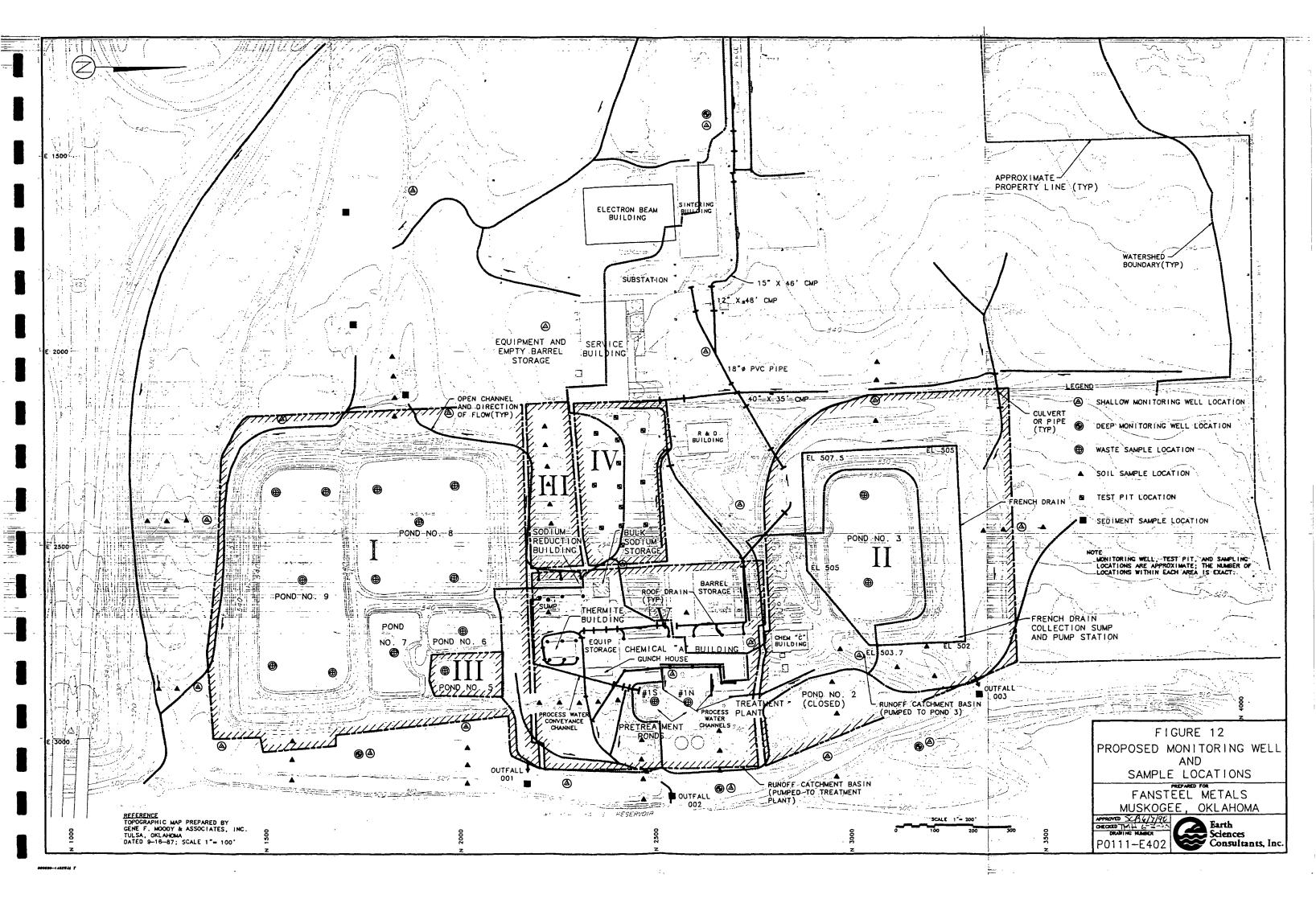


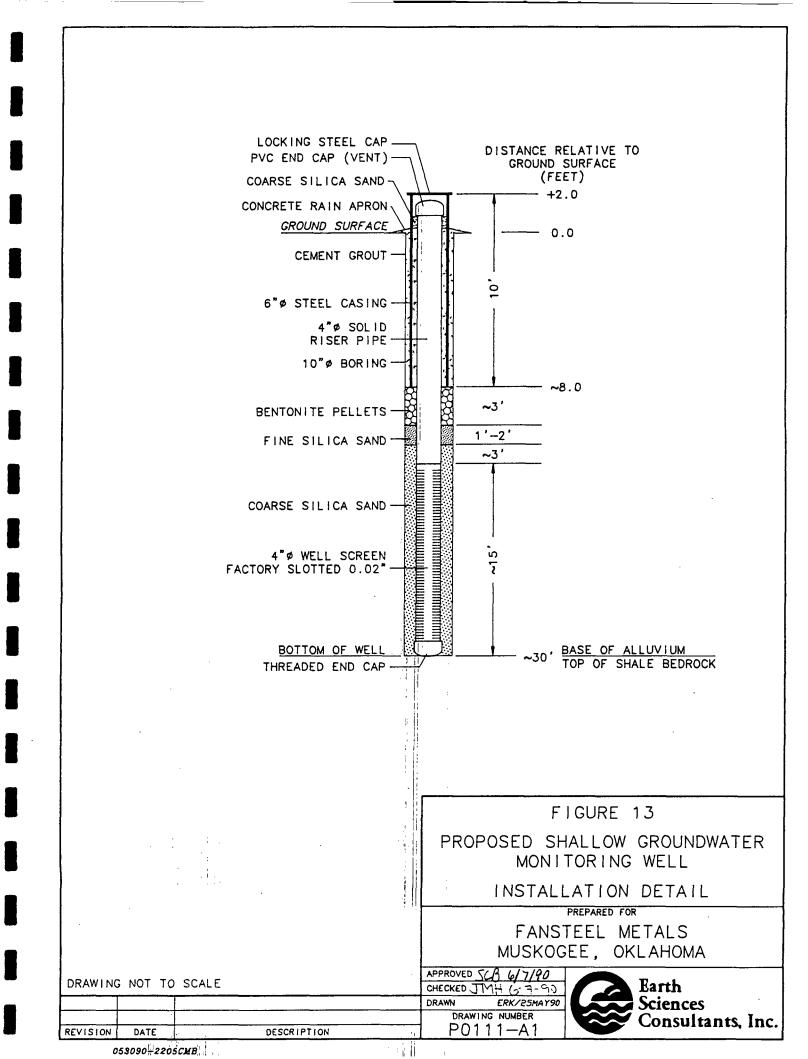


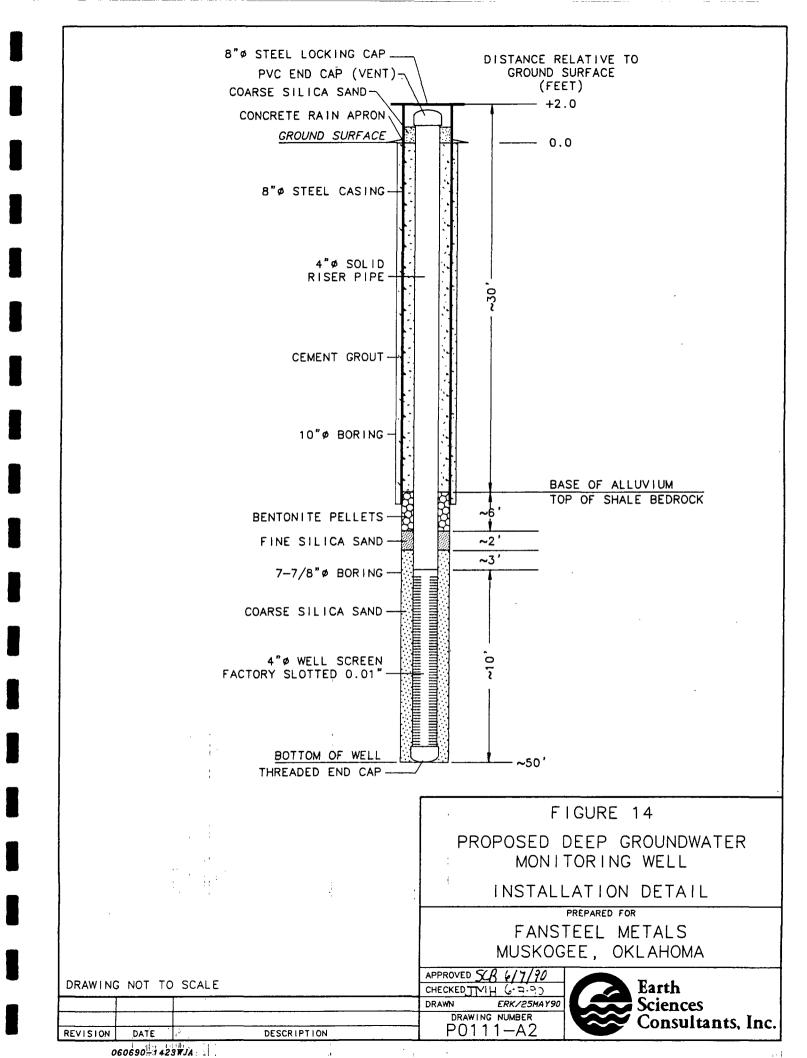




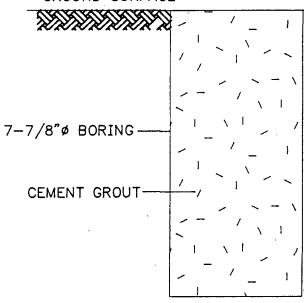








GROUND SURFACE



#### **NOTES**

REVISION

- 1. REMOVE ALL WELL SCREEN AND RISER PIRE AND REAM BOREHOLE WITH A 7+7/8 BIT.
- 2. ALL CEMENT GROUT TO BE PLACED FROM BOTTOM TO TOP OF BOREHOLE THROUGH TREMIE PIPE.

DESCRIPTION

DRAWING NOT TO SCALE

FIGURE 15

PROPOSED WELL ABANDONMENT DETAIL

PREPARED FOR

FANSTEEL METALS MUSKOGEE, OKLAHOMA

APPROVED SCB 6/7/90

CHECKED JMH 6-7-90

ERK/25MAY90

DRAWING NUMBER P0111-A3



053090-2306CMB

# FIGURE 16

# REMEDIAL ASSESSMENT PROJECT SCHEDULE WEEKS AFTER APPROVAL OF WORK PLAN

78 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 **TASK** 1. PROJECT MOBILIZATION 2. FIELD ACTIVITIES. (BORING AND MONITORING WELL INSTALLATIONS, GROUNDWATER, SEDIMENT WASTE AND SOIL SAMPLING, AIR MONITORING, WELL ABANDONMENTS). 3. ANALYTICAL 4. EVALUATION OF DATA AND REPORT PREPARATION 5. FANSTEEL AND REGULATORY MEETINGS

APPROVED SCB 6/7/40
CHECKED JMH (-7-96
DRAWN TPM/3IMAY90
DRAWING NUMBER
P0111—A4



# Appendix A

Quality Assurance Project Plan/ Sampling and Analysis Plan

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# Appendix A Quality Assurance Project Plan Fansteel Metals Muskogee, Oklahoma

#### A.1.0 Project Description

This Quality Assurance Project Plan (QAPP) describes the quality assurance/quality control (QA/QC) procedures that will be used for conducting the first phase of the remedial assessment at the Fansteel Metals (Fansteel) manufacturing facility in Muskogee, Oklahoma. The QAPP will address administrative, field, and laboratory QA/QC procedures and concerns.

#### A.1.1 Site Location and Description

#### A.1.1.1 Location of the Facility

The Fansteel site is located near the town of Muskogee, Oklahoma between Oklahoma State Route 165 and the west bank of the Arkansas River at River Mile 395. Federal Route 62 bounds the site to the south. The property consists of 110 acres of which approximately 40 acres are involved in the manufacturing processes carried out at the facility. The site assessment will address the entire 110 acres. The location of the site is shown in Figure 1. The layout of the site and the area to be addressed by the QAPP is delineated in Figure 2.

#### A.1.1.2 <u>Description of the Facility</u>

The manufacturing facility consists of several buildings, tank farms, storage areas, a wastewater treatment facility, active settling ponds, and inactive settling ponds. Of these areas and structures, the following will be subjects of the remedial assessment:

- o Service building
- o Equipment and empty-barrel storage area behind the service building
- o Storage pads
- o Ore storage area between the sodium reduction building and the "Thermite" building

- o Chem "A" building
- o Chem "C" building
- o Treatment plant
- o Outfall structures
- o R&D building
- o Tank farm area
- o Pond No. 3 and adjacent areas
- o Pond No. 5
- o Pond No. 2 (closed)
- o Ponds No. 6 through 9

In addition to these designated features which will be sampled or measured in some way, other areas will also serve as sources of samples to delineate background conditions and the limits of any possible soil, surface water, or groundwater contamination. Sample locations for soil, surface water, sediments, groundwater, and waste materials are shown in Figure 3.

#### A.1.2 Processes and Wastes at the Site

#### A.1.2.1 Process Description

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The Fansteel facility was used for approximately 30 years for the production of tantalum metal and columbium oxide from ores and slags. The manufacturing process involved digestion of the ore or slag in hydrofluoric acid in the Chem "C" building followed by extraction of the metal values from the digestion liquor with hexone (methyl isobutyl ketone, MIBK) followed by liquid/liquid extraction to separate the dissolved tantalum from dissolved columbium. The separate extracts were processed to the finished product metal and oxide in the Chem "A" building. Ore residue, spent hydrofluoric acid, and MIBK were sent untreated to Pond No. 3. Wastewaters from the processing of the tantalum- and columbium-bearing liquids were neutralized with slaked lime and the excess fluorides precipitated as calcium fluoride. The treated wastewater and neutralization sludge reported to Ponds Nos. 6 through 9 for settling before eventual discharge through Outfall 001 to the Arkansas River.

Pond No. 3 is not the only structure to have been used to contain production residues. The closed structure, Pond No. 2, served the same function as Pond No. 3. There are two abandoned residue ponds, "old" Pond No. 3 and a Pond No. 4 under the existing Pond No. 3 structure. The contents of these ponds may have been removed in whole or in part in the construction of the new Pond No. 3. Pond No. 5 in the wastewater settling ponds area also contains process residues.

Other waste disposal or management practices are believed to have taken place during the lifetime of the plant. Drums may have been buried behind the service building and/or below the process residues in Pond No. 5. Contaminated soils have been stored between the sodium reduction building and the "Thermite" building.

The tantalum- and columbium-containing ores and the tin slag (also used as a source of tantalum and columbium) contain small but significant amounts of naturally occurring uranium and thorium. Process residues, areas subject to dust generated from ore and slag milling, areas used for ore or slag storage, and areas affected by process upsets may exhibit elevated levels of radiation. Radioactivity may also be present in the groundwater and wastewater treatment residues.

The site assessment was triggered by a release of material from Pond No. 3 in June of 1989. Approximately 90,000 gallons of liquid from Pond No. 3 escaped due to a liner failure. Some of the liquid escaped into the Arkansas River; some of it may have seeped into the ground and reached at least the upper aquifer. A substantial body of soil is probably contaminated. Since the released liquid had to be disposed, this radioactive solution was also run through the treatment plant. This may have resulted in contaminating some or all of the wastewater settling ponds with radioactive materials.

Since the release, Fansteel has shut down the tantalum and columbium extraction portion of the plant and does not plan to reopen it. The presence of uranium and thorium in the ores necessitated obtaining a license from the Nuclear Regulatory Commission (NRC) for the possession of nuclear source material. Because the facility is being shut down, it must be formally decommissioned. Many portions of the site assessment will be used in support of the facility decommissioning

plan. In addition, decommissioning plan monitoring requirements that might not normally be addressed in a site assessment to delineate environmental effects will also be performed. This QAPP will, therefore, be concerned with the data quality requirements of the NRC as well as the U.S. Environmental Protection Agency (USEPA).

#### A.1.2.2 Chemicals Used and Wastes Produced

Unlike uncontrolled waste disposal sites or sites that may have received a variety of wastes from a number of sources, all of the manufacturing processes and waste materials that were ever produced or employed at this site are known. There have been no substantial changes in the manufacturing processes used throughout the life of the facility. This knowledge has a significant bearing on some of the QA/QC procedures that will be appropriate or applicable for the conduct of the site assessment and decommissioning plan survey.

The chemicals and wastes listed below were used in or produced by the facility. Any of the chemical raw materials or their reaction products may show up in the process wastes. Furthermore, any unused chemical raw material that is discarded or used in a manner constituting disposal must be considered a waste as well. Following are these chemicals and wastes:

- o MIBK
- o Hydrofluoric acid
- o Sulfuric acid
- o Potassium fluoride
- o Sodium hydroxide
- o Sodium metal
- o Sodium chloride
- o Ammonia
- o Quicklime (calcium oxide)
- o Slaked lime (calcium hydroxide)
- o Nitric acid
- o Hydrochloric acid
- o Tantalum/columbium ore
- o Ore digestion residue (waste)
- o Wastewater treatment plant residue (waste)

No other chemicals were used in significant quantities at the facility. Materials used in service and maintenance of plant equipment (i.e., cleaning solvents, paints, and lubricants) would have been present in nondetectable quantities in

the treatment plant residues if they were discarded by this route. There is no evidence of improper disposal or management of these materials.

#### A.1.3 Study Objectives and Sources of Data

#### A.1.3.1 Use of Data to be Acquired

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This site assessment will be performed to provide the answers to several questions concerning the Fansteel site. Some of these relate specifically to the consequences of the June 1989 release; others to general facility contamination by process chemicals and radionuclides. Specifically, the data must be able to support conclusions with regard to the following issues:

- o Determine if there is groundwater contamination traceable to site operations.
- o Determine if there is groundwater contamination as a result of the June 1989 release from Pond No. 3.
- o Determine extent of surface soil contamination both vertically and horizontally as a result of the June 1989 release.
- o Determine the presence and extent of other chemical contamination of surface soils.
- o Determine the presence or absence of buried containers of waste on the site.
- o Determine the quantities of ore digestion waste at the site.
- o Determine the quantities of wastewater treatment plant residues at the site.
- o Determine the degree of homogeneity of the ore digestion waste and wastewater treatment plant residues, particularly in the vertical plane.
- o Determine the chemical properties of the digestion and water treatment plant wastes.
- o Determine the presence and extent of subsurface soil contamination.
- o Determine the presence and extent of surface water and sediment contamination.

- o Determine the extent of radiochemical contamination in buildings, structures, soil, subsurface materials, groundwater, and waste materials.
- o Determine if contamination is leaving the site via surface drainage, groundwater migration, or through the air.
- o Determine the degree of hazard posed by the site to the environment and to public health.
- o Support the conduct of the feasibility study.
- o Support the production of an acceptable decommissioning plan for the portions of the site licensed by the NRC.

These objectives will be achieved by a program of environmental sampling and monitoring, geophysical surveys, and waste sampling supported by laboratory analysis.

#### A.1.3.2 Data Acquisition

Data will be obtained from a series of field instrumental measurements, visual observations, field chemical measurements, and laboratory analysis of environmental and waste samples. Samples and measurements will be taken from defined locations and depths so that the information obtained can be related to site conditions. The details of sample types and the sample locations are presented in the work plan.

#### A.1.3.2.1 Groundwater Samples

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Existing monitoring wells are insufficiently documented. Thus, historical data can only be used as a general water quality indicator.

Additional monitoring wells will be installed on the site. These well locations are also found in Figure 3. Three of the wells will be deep (i.e., finished in the consolidated material underlying the site). The other wells will be shallow, drawing water from the unconsolidated alluvium. There will be four overburden wells and one bedrock upgradient well which should yield background water quality data. The others are detection wells for determining the presence and extent of groundwater contamination. A detailed record of well installation and materials encountered during well installation will be made by a qualified field geologist.

Instrumental measurements for organic vapor and radioactivity will be conducted on the materials brought to the surface during installation of the wells.

#### A.1.3.2.2 Surface Water Samples

Surface water leaving the site will be sampled just before it enters the Arkansas River. One sample will be obtained from the small stream that passes just to the south of the wastewater settling ponds. This stream receives no effluent from the facility. Runoff from the developed portions of the property is diverted away from this stream as well. Elevated levels of any of the chemicals used in the facility may indicate systemic site contamination.

For comparison, there will also be two water samples drawn from the Arkansas River. One sample will be upstream of the plant site; the other downstream.

#### A.1.3.2.3 <u>Effluent Samples</u>

Water samples will be obtained from all three outfalls. These samples will be taken when the outfall is discharging under normal conditions of operation. In the event of any upsets during the course of the remedial assessment, additional samples will be taken from the affected outfalls.

Although the manufacturing facility is no longer in operation, the plant's wastewater treatment facility is still in use, so Outfall 001 normally has a discharge. Outfalls 002 and 003 discharge in response to weather conditions.

#### A.1.3.2.4 Surface Soil

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Surface soils will be obtained at the locations shown in Figure 3. All soil sampling activities will be accompanied by field instrumental survey equipment. This equipment will consist of a suitable organic vapor detector and a radiation detector capable of responding to alpha-particle radiation. Results of the field monitoring will be recorded for each sample site. Surface soil samples will consist of the top six inches of material, obtained in such a manner as to preserve a uniform representation of the soil column. A detailed log of soil visual characteristics will be prepared at the time of sampling.

#### A.1.3.2.5 Subsurface Materials

Surface soil sample locations will also serve as the sites for sampling subsurface materials. These samples will be obtained using a split-spoon device. Spoons will be scanned for radioactivity and presence of organic vapor immediately upon opening. A detailed descriptive log of visual characteristics will be made.

#### A.1.3.2.6 Waste Materials

Waste materials will consist of the solid, liquid, and semisolid residues in Pond No. 3 and the wastewater treatment ponds. Any other waste materials that may be encountered will also be sampled. This would include any buried wastes, materials from the closed process waste impoundments, ores, slags, leftover process or maintenance chemicals, etc. Appropriate field descriptions and instrument measurements will be made for each type of waste encountered.

#### A.1.3.2.7 Radiation Samples

Building interiors and outside areas that may reasonably be expected to have been affected by radioactive contamination will be examined with appropriate detection instruments. Wipe samples or samples of accumulated dust will be obtained for radiological analysis. The locations of these sample points is not known at this time. Target areas of concern are the Chem "A" building, Chem "C" building, R&D building, ore storage pads, and associated outside areas. However, general site scanning for elevated radioactivity will also take place.

#### A.1.3.2.8 Test Pit Excavations

Test pits will be dug in areas that may have been used for burial of drums or other containers of waste. The location of the test pit areas is shown in Figure 3. Test pits will be logged and examined for the presence of organic vapors and elevated radioactivity. Soil samples will be taken for chemical or radioactivity analysis if field instruments indicate the presence of foreign materials.

#### A.1.3.2.9 Geophysical Surveys

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There are many areas of the plant that are not suitable for a surface geophysical survey because of the amount of metals around in tanks, pipelines, and structures. However, if such a survey can be conducted to detect the presence of

buried metal items (e.g., drums, unmapped tanks, etc.), it will be carried out. Findings from this survey (if positive) will be used to guide future sampling operations and other explorations.

#### A.1.3.2.10 Ambient Air Monitoring

Five air-monitoring stations will be set up on the site at the locations shown in Figure 3. These stations will be used to collect particulate samples from upwind and downwind locations to determine whether or not waste constituents are leaving the site.

#### A.2.0 Project Organization and Responsibility

This project will be managed by Earth Sciences Consultants, Inc. (Earth Sciences). The owner/operator of the site and concerned state and federal regulatory agencies may appoint persons to review and approve portions of the project plans. We do not envision anyone from outside the Earth Sciences organization having project or operations management responsibilities unless specifically delegated by the Earth Sciences project manager.

The following personnel or designated positions will be assigned to this project. If key personnel are changed during the course of this project, this and other affected documents will be suitably amended.

#### A.2.1 Earth Sciences Personnel

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The following Earth Sciences personnel will be involved in project management or oversight positions:

- o Project Director Scott C. Blauvelt will be responsible for the overall management of all phases of the project.
- o Project Manager Joseph M. Harrick will be responsible for development and implementation of all work plans relating to this project.
- o Project Coordinator Peggy L. Carpenter will insure the orderly progression of work activities and maintenance of proper documentation to support remedial assessment goals.

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- o Site Supervisor <u>(to be determined)</u> will be responsible for day-to-day operations at the site. He will be responsible for making sure that all tasks are completed to the specifications contained in the work plan.
- o Quality Assurance (QA) Coordinator Paul N. Taylor will be responsible for the development of QA procedures for project activities and for the review and maintenance of the documentation of project activities.

#### A.2.2 Antech Ltd. Personnel

Laboratory services will be provided by Antech, Ltd. (Antech) for the analysis of chemical parameters on soil, water, waste, and air samples. The following Antech personnel will be responsible for scheduling and performance of laboratory analysis:

- o Laboratory Manager David M. Miller will be responsible for all activities relating to scheduling and performance of required analyses.
- o Analytical Group Leaders Aaron D. McGee, Kathryn L. Stoudnour, Carl A. Martin, and John M. Smith are responsible for the timely performance of analyses by the specified methods in their areas.
- o QA Coordinator Mary Ellen Tenney will be responsible for insuring that all required quality control samples are run, that all documentation relating to analyses performed for this project is complete, and that the requirements of the specific methods have been met.

### A.2.3 <u>Contractor Laboratory for Radioactive Materials Analysis</u> (To be determined)

#### A.3.0 QA Objectives for Project Data

The project QA objective is the generation of sufficient data to produce a remedial assessment report. The remedial assessment report, in turn, will enable the project staff to determine the extent of environmental contamination and choose an effective remediation strategy (if necessary). This objective applies both to field operations and laboratory activities.

#### A.3.1 <u>Data Quality Objectives - Stage II</u>

A semiquantitative analysis of data quality objectives was performed for this initial phase of the remedial assessment. The project does not involve an investigation of a site with unknown properties or contaminants. Probable source areas are well defined and the possible contaminants are well characterized. Therefore, in this preliminary phase, a rigorous statistical sampling plan is not needed.

#### A.3.1.1 Identify Data Uses

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The first phase of the investigation will seek preliminary answers to the questions and issues enumerated in Section A.1.3.1 of Chapter A.1.0. The following specific determinations will be made using data from the first phase of the remedial assessment:

- o Presence and location of areas of groundwater contamination in the upper aquifer.
- o Presence of contamination in the lower aquifer.
- o Presence of soil contamination in the area affected by the June 1989 release from Pond No. 3.
- Presence of soil contamination in other manufacturing areas.
- o Presence of buried containers in areas suitable for surface geophysical surveys.
- o Determination of the quantities of wastewater treatment sludge and residue in Pond No. 3.
- o Presence of sediment and surface water contamination.
- o Presence and extent of radiochemical contamination in and on structures and equipment in the plant area.
- Presence of radiochemical contamination in other portions of the facility.
- o Presence of airborne chemical and radiochemical contamination around the facility perimeter.

The sampling plan for Phase I operations may also yield information on the extent of contamination present on site. However, if a pollutant boundary is not determined during the first phase of the investigation, a second round of

sampling will be conducted. The spatial distribution of the second sampling round will be determined by a statistical analysis of data quality objectives.

The sludge sampling will be performed according to a statistical plan to determine the average value of critical constituents. The wastewater treatment ponds and Pond No. 3 will be sampled on a 50-foot grid through the entire sludge column. Each individual column will be composited but each grid point will be treated as a separate sample.

The data developed during this phase will be used to confirm or modify the assumptions made in the Health and Safety Plan (HSP) for site operations. The findings will also be used in the development of the HSP for remedial operations should they be necessary.

The data from the first phase of the remedial assessment will be used in conducting the site risk assessment (RA). While the data developed in this phase may not be sufficient to complete an RA, they must be of sufficient quality for inclusion with other data that may be developed at a later time.

#### A.3.1.2 Data Types

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Samples will be obtained of each of the following materials:

- o Groundwater
- o Surface water
- o Surface soil
- o Subsurface soil
- o Digestion sludge
- o Wastewater treatment sludge
- o Plant point source effluents
- o Ambient air
- o Building surfaces
- o Any other waste or wastelike materials that may be discovered or encountered

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Background samples will be obtained for all of the environmental media. Soil, deep and shallow groundwater, air, and surface water monitoring points have been chosen that should provide uncontaminated background data.

A certain percentage of the samples of environmental materials will be analyzed for the entire Target Compound List (TCL) of chemical parameters. All the samples of environmental materials will be analyzed for the following contaminants known or believed to be present:

- o Tantalum
- o Columbium
- o Radioactivity (gross alpha and gross beta)
- o MIBK
- o Fluoride
- o Total calcium
- o Total sulfate
- o Total metals
  - Lead
  - Nickel
  - Antimony
  - Arsenic
  - Barium
  - Cadmium
  - Chromium
  - Mercury
  - Selenium
  - Silver
- o Hydrogen ion activity (pH)
- o Ammonia nitrogen

In addition, 20 percent of solid materials samples will be tested by the Toxicity Characteristic Leaching Procedure (TCLP) for the following metals:

- o Arsenic
- o Barium
- o Cadmium
- o Chromium
- o Lead
- o Mercury
- o Selenium
- o Silver

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This will determine the mobility of any of these contaminants detected. Samples of waste materials will be analyzed for the constituents likely to be present. In the case of the Pond No. 3 residues and similar materials, they will be

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assayed for tantalum and columbium metal values (i.e., percent composition rather than parts per million as contaminants). A portion of the samples will also be assayed for the amount of uranium and thorium present. The Pond No. 3 residue and similar materials are potential commercial products.

Air samples will be analyzed for total suspended particulates, fluorides, and radioactive species. If radioactive constituents above background are detected, the contributing species will be identified.

Some physical properties will also be measured in this first round of data collection. These data include the following:

- o Groundwater elevations
- o Soil and subsoil profiles
- o Descriptions of any wastes encountered
- o Percent solids composition of sludges and residues
- o Surface and removable radioactivity

#### A.3.1.3 Data Quality Needs

Analytical laboratory data must be of adequate quality to determine remediation needs or alternatives. Field location of data points must be within the limits of normal land survey practices. Accurate field screening data will guide in the selection and use of personal protective equipment and detect organic vapors, conductivity, pH, and radioactivity as indicators of contamination. For the building and equipment radiation survey, the instruments must be capable of determining whether or not the NRC standards for release for unrestricted use have been met at an acceptable level of confidence.

#### A.3.1.3.1 Level I Data Needs

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Level I data will be provided by field screening of environmental and waste material samples for clear evidence of contamination. Examples of Level I data include the results of organic vapor analyzer (OVA) screening of recovered soil, subsurface material, waste, and liquid headspace. Positive readings strongly indicate the presence of contaminants from the ore digestion process. Also, field screening for radioactive materials will indicate semiquantitatively the amount of radionuclides that may be present. This information is used for guiding further sampling activities and for establishing health and safety

protection levels. Screening activities will take place at all sample locations and will be performed on all samples.

#### A.3.1.3.2 Level III Data Needs

Most of the samples obtained in this round will be subject to Level III data quality requirements. Where procedures approved by USEPA exist, they will be used. The procedures' QA/QC requirements will be met in full but Contract Laboratory Program/Routine Analytical Services (CLP/RAS) documentation packages will not be routinely provided. All routine chemical analyses will be performed to Level III standards except samples subject to the full TCL analytical suite, tantalum and columbian analysis, radioactivity analysis, or radionuclide identification.

#### A.3.1.3.3 Level IV Data Needs

Level IV data are chemical analyses performed to the standards of the CLP/RAS. This level of data will be required for a portion of the groundwater and soil samples be subject to TCL analysis. Based on the current site model, these samples will be used to document the absence of contaminants other than those identified in connection with known manufacturing processes. If the samples subject to TCL analysis fail to show contaminants other than those anticipated, no further Level IV data will be collected during the site assessment.

#### A.3.1.3.4 Level V Data Needs

Because unconventional materials will be analyzed, there will be a significant amount of Level V data. Level V analyses are nonstandard methods for constituents for which there are no USEPA-approved methods or matrices in which the USEPA-approved methods do not function adequately. Examples of Level V data that will be generated include analysis of tantalum and columbium, uranium and thorium, and removable radiation counting.

#### A.3.1.3.5 Contaminants of Concern

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From an environmental protection viewpoint, most of the chemicals associated with the manufacture of tantalum metal and columbium oxide are deleterious and many are on the Comprehensive Environmental Response, Compensation, and Liability Act list of environmental contaminants. In order to prevent degradation of the

environment, hazardous substances that may be present need to be identified, quantified, and (if possible) removed or immobilized.

Certain of the contaminants which may be present would present public health hazards of distinct importance. Chief among these are the radionuclides and their daughter products (which are inhalation and ingestion hazards), inorganic fluorides, and hydrofluoric acid.

The most significant and pervasive indicator parameters for contamination at this site will be the fluoride-ion and radioactive materials. These substances are found throughout the manufacturing process and may occur in any of the environmental media. The analytical tests for these species are well developed and can be rapidly performed. Some radiological analysis can be performed on the site.

#### A.3.1.3.6 <u>Detection Limits Requirements</u>

For all TCL substances analyzed at quality Level III or Level IV, the project's detection level will be at the USEPA's contract-required detection level for CLP work. For the determination of tantalum, columbium, uranium, and thorium, the detection levels in water will be 100 parts per billion, 50 parts per billion, 1.0 part per billion, and 10 parts per billion, respectively. In soil, the detection limits will be 10 parts per billion, 5 parts per billion, 1 part per billion, and 1 part per billion, respectively. For detection of surface radio-activity on building surfaces and on equipment, the instruments used will be able to detect ten counts per minute above background. The counter for determination of removable radioactivity will be able to detect one count per minute above background.

#### A.3.1.4 Data Quantity Needs

The amount of information on the site's previous operations was used to select the data density for this first phase. Based on the findings from Phase I of the remedial assessment, further data needs can be evaluated. This first round of data points was chosen on the basis of knowledge of previous operations and their locations. The chosen points are ones most likely to show evidence of contamination if contamination is present.

#### A.3.1.5 <u>Sampling and Analysis Options</u>

For the first round of analyses, all designated environmental samples will be obtained as quickly as possible and submitted for laboratory analysis. All samples will be subject to field scanning but will also be subject to laboratory analysis regardless of the field-screening findings.

Waste samples (i.e., contents of the wastewater treatment ponds, Pond No. 3, Pond No. 2, and Pond No. 1S) will be subject to close visual examination as the materials are removed from the sampling device as well as instrumental monitoring for organic vapors and radioactivity. If unexpected inhomogeneity is observed or if a single core cannot be obtained through the depth of the material, an alternative method (i.e., use of a vibrating sectioned corer) will be used. Additional samples of visibly different materials may be taken based on field observations.

### A.3.1.6 <u>Precision, Accuracy, Representativeness, Completeness, and Comparability Evaluation</u>

Precision, accuracy, representativeness, completeness, and comparability (PARCC) evaluation can be rigorously applied to laboratory analytical products. It is a less-useful technique for evaluating field activities, especially for a preliminary site assessment.

#### A.3.1.6.1 Precision Requirements and Expectations

Laboratory precision (reproducibility) will be that required by the individual analytical methods as established under the current CLP statement of work (SOW). The SOW requirements will apply whether data are presented as Level III or Level IV. This precision will be demonstrated by the laboratory both by historical replicate measurements and by replicate measurements on a portion of the samples submitted for analysis. This portion will be at least 1 sample of each matrix or 1 sample in 20 or a fraction thereof (if more than 20 samples of a given matrix are obtained). These will be blind replicates. The laboratory will also perform duplicate analyses according to its own protocols.

Sampling precision is more difficult to evaluate. Soil and sludge materials samples can be taken close by each other; however, the inherent anisotropy of these materials is such that no firm conclusion could be drawn from analytical

data generated from these samples whether the analyses were in close agreement or not. With water samples, where the material may be presumed homogeneous, replicate samples will be obtained to demonstrate the precision of sampling activities. Two field groundwater samples will be taken in replicate as a check on precision.

#### A.3.1.6.2 <u>Accuracy Requirements and Expectations</u>

Accuracy of laboratory determinations is determined by the recovery of known-value samples and spiked samples. Accuracy of all Level III and Level IV analysis samples will be to the standards established by the current CLP SOW. All work involving the production of knowns and spikes will be performed in the laboratory by laboratory personnel.

Accuracy is less meaningful in terms of sample acquisition. One field task that is subject to reasonable accuracy measurement is the determination of sample locations and well sampling depths. Wells and other sample points will be located to within one-quarter foot ( $\pm 3$  inches) in the horizontal plane. Top-of-casing elevations will be determined to within  $\pm 1$  inch. Sample depths will be located within  $\pm 1$  inch or  $\pm 0.1$  foot.

#### A.3.1.6.3 Representativeness Requirements and Expectations

For the preliminary round of sampling activities governed by this QAPP, it is not anticipated that the results will be representative of the site conditions as a whole. In fact, the data points are chosen in an attempt to intercept exceptional nonrepresentative conditions (i.e., the presence of contaminants in the soil or groundwater).

We do, however, expect that the samples acquired will be representative of the locations sampled. To insure this, standard operating procedures have been developed to guide all sampling activities as well as preservation, transportation, and documentation. Representativeness is insured in the laboratory operations by the strict adherence to the published methods for sample preparation and analysis.

#### A.3.1.6.4 <u>Completeness Requirements and Expectations</u>

Laboratory data that are generated at Level IV will have to demonstrate a minimum of 85 percent completeness in the data validation report. Level III data will be audited for completeness in accordance with the method requirements. A completeness of 80 percent will be required for these data. For these standards to be met (85 percent and 80 percent, respectively), all samples must be 100 percent complete in terms of the data validation requirements.

Field documentation of sample acquisition operations, well installation, and other activities will be audited. A completeness record of at least 75 percent will be required for operations to be considered satisfactory.

#### A.3.1.6.5 Comparability Requirements and Expectations

It is anticipated that more than one round of sampling activities will take place. It is also anticipated that data developed in the course of the site assessment will be used in conjunction with and in support of the facility decommissioning plan. Therefore, comparability from one set of samples to the next is vitally important. Comparability depends on the other components of the PARCC evaluation remaining in control and consistent across data sets. An ongoing program of auditing, review, and laboratory QA/QC will be undertaken to insure that comparability is maintained.

#### A.3.2 Field QA Requirements

Field measurements and sampling activities will be documented in such a way as to unambiguously identify the following as applicable:

- o The activity performed
- o The person or persons performing the activity
- o The location of the activity
- o Time and date
- o Weather conditions
- o Instruments or devices used
- o Procedures employed
- o Measurement results

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- o Sample identification
- o Any relevant or required observation

To the extent that Earth Sciences has a standard form for an activity, the standard form should be used. For tasks that do not have a standard form, all

observations and other required information should be entered into a field book. Field notebooks should have each page dated and initialled.

The Earth Sciences QA coordinator will conduct periodic reviews of field records to insure that the required level of information is being recorded and that proper format is used. The QA coordinator will also be responsible for the review or creation of specific standard procedures for field activities where needed.

#### A.3.3 <u>Laboratory QA Requirements</u>

The laboratory will meet the QA requirements specified in the analytical methods. The individual methods must be consulted for the required standards of accuracy and precision, number of spiked samples, frequency of replicates, etc. Chemical analysis will be performed in accordance with the requirements of SW-846 for all parameters with methods found in this reference. If other parameters are to be analyzed for, appropriate accepted published methods will be used.

#### A.3.4 Engineering QA Requirements

Engineering QA primarily involves peer review of all calculations, drawings, and conclusions based on interpretation of data. All calculation sheets, etc., are checked and signed off by a person capable of performing the calculations or otherwise familiar with the materials. Conceptual engineering QA is informally established by consensus within the project staff.

#### A.4.0 Sampling Procedures

Sampling of environmental materials (i.e., soil, groundwater, surface water, subsurface materials, and air) will be done in accordance with the standard operating procedures attached to this QAPP. Solid waste materials will generally be sampled as though they were soil or soil-like materials. Semiliquid materials such as the treatment plant settling pond sludge will be sampled using a staged "vibra-core" device or other effective means to obtain representative samples through the depth of the material.

#### A.4.1 Parameters and Measuring Systems

#### A.4.1.1 Analytical Parameters

Under this plan, 30 percent of the groundwater samples will be analyzed for the entire TCL of the current CLP SOW. In addition, all samples except air will be analyzed for the following parameters:

- o TCLP leachable metals
- o TCLP total digestible metals
- o MIBK
- o Total calcium
- o Total fluoride
- o Total sulfate
- o Ammonia nitrogen
- o Gross alpha radioactivity
- o Gross beta radioactivity
- o pH

Air samples will be analyzed for total suspended particulates, gross alpha radioactivity, and gross beta radioactivity. If downwind samples exceed upwind samples in radioactivity by a factor of 2 or more, the air filter will be analyzed for uranium and thorium. If the downwind total suspended particulates exceeds the upwind samples by a factor of 2 or more, the particulates will be analyzed for total fluoride and the TCLP total metals.

Waste samples from Ponds Nos. 2, 3, and 5 and any other materials that may substantially contain digestion process residues will be assayed for uranium, thorium, tantalum, and columbium metal values. This action is not in itself a part of the site assessment. These materials are potential commercial products if their metals values are sufficiently high.

#### A.4.1.2 Pollutant Measuring Systems

Most of the pollutant measuring systems consist of laboratory instruments. The laboratory instruments that will be used, apart from sample preparation and extraction equipment, will or may consist of the following:

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- o Gas chromatograph/mass spectrometer
- o Gas chromatograph with electron capture detector
- o Gas chromatograph with photoionization detector
- o Gas chromatograph with flame ionization detector

- o Atomic absorption spectrophotometer
- o Inductively coupled plasma spectrophotometer
- o Multiple ion analyzer
- o Radiation counter for alpha and beta detection

Field pollutant measuring systems will consist of: (1) an array of monitoring wells for obtaining groundwater samples from identified water-bearing formations and (2) an array of monitoring devices for obtaining direct readings of certain important values in a real or near-real time mode. Airborne contaminants will be trapped using high-volume air filters for total suspended particulates. The portable instruments will consist of the following:

- o OVA with photoionization detector
- o OVA with flame ionization detector
- o pH meter
- o Conductivity meter
- o Radiation detector for beta particles
- o Radiation detector for alpha particles

#### A.4.2 Sample Site Selection Criteria

Some sample point locations are determined by the site itself. Other sample location selections are not predetermined. The criteria for selecting the sites mapped in Figure 3 are presented in the following sections.

#### A.4.2.1 Stream Samples

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If the small stream at the southern end of the property is sampled, it will be sampled as close to the mouth of the stream as practicable. Sampling will be done when the stream is flowing at a rate high enough to allow a good sample to be obtained. However, the sample will not be taken immediately after a substantial rainfall. The water sample should be representative of baseflow conditions.

Samples may also be obtained from the Arkansas River during later stages of this study. The samples from the Arkansas River will be taken in a manner such that they will be representative of river water quality but will also detect any contributions from the facility. Samples will be taken from two or three feet below the water surface. If there are any seeps along the riverbank, they will be sampled at some time when they are adequately discharging.

#### A.4.2.2 Groundwater Samples

The proximity of the river will probably control the flow of groundwater. Previous monitoring data of uncertain value indicate that the direction of flow is generally toward the river and that bank discharge is the normal case. This establishes the east side of the property as downgradient and the west side as upgradient.

The shallow monitoring wells have been located close to potential sources of contamination. Thus, the Pond No. 3 area and the wastewater settling ponds are surrounded by monitoring wells. There is also a monitoring well on the downgradient side of the Chem "A" building. Other wells are located along the riverbank and along the upgradient side of the facility. If there is shallow groundwater contamination from the facility, these wells are located to detect that contamination as well as to determine its probable source area. The upgradient wells are located far enough to the west that they should not be affected by activities at the facility.

The deeper wells are located in a general detection pattern of one upgradient and two along the riverbank. These wells are located in order to be able to detect the presence of contaminants in the lower aquifer. There are not enough deep wells to locate the source of any contamination that may be found. Since there is no reason to believe that the upper aquifer in the unconsolidated material and the bedrock aquifer are not interconnected, the shallow wells will probably be adequate to locate the source of any contamination found in the deeper wells.

#### A.4.2.3 Outfalls

Outfalls will be sampled at the discharge point when they are discharging normally. Facility personnel conduct this sampling as part of the discharge permit compliance monitoring program.

#### A.4.2.4 Soil and Subsurface Materials

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Soil sample locations were chosen in areas most likely to show effects of contamination from facility operations. No attempt was made to conduct random soil sampling because of our level of knowledge of the plant processes and operating history. Soil samples will be taken in radial patterns from the Pond No. 3 and treatment plant settling pond locations. The old ore storage area will

be sampled densely as will the area along the riverbank downstream (and generally also downwind) of the plant. Samples for background determinations are located generally upwind of the facility and as far removed from the manufacturing operations as possible.

#### A.4.2.5 Sediment

Sediment samples will be taken from each outfall and from the stream channel south of the plant. The exact location of the samples cannot be specified in advance. Areas will be sought in the field that have encouraged the accumulation of sediments to determine if there is evidence of solid materials such as the ore and slag that were carried off site by surface runoff.

#### A.4.2.6 <u>Wastewater Treatment Sludge</u>

The contents of the wastewater treatment plant ponds will be sampled in 5-foot vertical increments on 50-foot centers. The entire sludge column will be sampled. Based on the results of the visual inspection and instrumental monitoring of the samples, a decision will be made concerning which samples should be submitted for analysis and which samples should be archived.

#### A.4.2.7 Pond No. 3 Sludge

Characterization of the Pond No. 3 sludge is particularly important since this material is a potential commercial product. Visual inspection of the exposed material indicates significant variability in the material both in the horizontal and the vertical planes. Sampling will be on a 50-foot grid. Vertical spacing of sampling will depend on visual observations made at that time.

#### A.4.2.8 Other Digestion Wastes

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Other deposits of digestion waste are found in the areas designated as Pond No. 2 and Pond No. 5. Other areas that may contain this waste are below the present Pond No. 3 and east of the Chem "A" building. These locations will be sampled to demonstrate the presence or absence of the digestion residue. Areas that are found to contain the residue will be sampled by a procedure similar to that discussed in Section A.4.2.7 (above) for Pond No. 3. (Areas below the liner of Pond No. 3 cannot and will not be sampled until the contents of Pond No. 3 and its liner have been removed.)

#### A.4.3 <u>Specific Sampling Procedures</u>

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Earth Sciences has developed specific procedures for sampling the materials that will be of interest at this site. These procedures are reproduced below.

#### A.4.3.1 Standard Procedures for Sampling Groundwater

Groundwater samples are obtained from wells or springs. The sample source of choice is a purpose-built groundwater monitoring well. A variety of tools are used in groundwater monitoring including several different types of pumps and bailers as well as depth-measuring devices.

#### A.4.3.1.1 Purging Monitoring Wells

Water sampled for chemical analysis must be representative of the water in the producing formation. Water that has been standing in the well casing for an extended period of time will be different from water in the surrounding formation. To obtain formation water, the well must be purged of water in the borehole and casing.

Depending upon the diameter of the well, total depth, and elevation of the water table, purging may be most efficiently performed using pumps or hand-operated bailers. No special care needs to be exercised to preserve labile components of the purged water. Therefore, impeller-driven submersible pumps or airlift pumps can be used for purging as long as no contaminants are added to the water by the use of the pump. Bailers may be used if depth and volume make their use efficient.

For most operations, an adequate purge is defined as either three well volumes or evacuation to dryness. Specific project requirements may specify some other purge quantity. A well volume is calculated using the following formula:

$$V = \pi r^2 (h_1 - h_2) (7.58)$$
 (1)

where

r = well radius in feet,

 $h_1$  - total well depth in feet, and

 $h_2$  = depth to water in feet.

It must be noted that  $h_1$  and  $h_2$  must be measured from the same reference point. Since most monitoring wells are constructed with the well casing protruding some height above the ground, normal practice is to use the top of the casing as the reference point.

There is disagreement over which well radius to use, radius of the well casing or the radius of the borehole. It is Earth Sciences' policy to use the borehole radius as the well radius rather than the casing radius since water in the annular space between the casing and the undisturbed formation is not (strictly speaking) formation water.

If a purpose-built monitoring well is being sampled, the well construction details should give the total well depth. Information reported by organizations other than Earth Sciences should be field checked prior to sampling a well. Depth to water must be measured each time any water is removed from a well, whether for purging or sampling.

If another well type (such as drinking water, stock, or process water) is being monitored or if well installation information is unknown or suspect, total depth must be measured using a weighted calibrated rope if access to the borehole is possible. Depth to water will be measured with an electrically actuated water level indicator.

The volume evacuated from a well is determined by actual volumetric measurement of water removed from the well or by measuring the rate and duration of pumping. If a well is being purged by bailing, the volume in a full bailer should be calculated or measured. Then a count of bailer volumes can be maintained to obtain a measure of the total volume removed. If a well is evacuated to dryness, a record of the total volume removed should be made. Upon completion of the purge, the water level should be measured again.

#### A.4.3.1.2 Purging Other Types of Wells

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Wells developed for water supply may not have ready access for direct measurement of water level or well depth. Available information should be used to make a best conservative estimate of well volume. Sources of information and values used should be reported. Water supply wells may have permanently mounted pump

systems that must be used for removal of water. Be sure to exhaust purge water upstream of any holding tank or in-line water treatment system is possible.

Piezometers are wells, usually of the narrowest practical diameter, used primarily for water-level measurements. They can also be used to obtain samples for groundwater quality determinations. Purging procedures for piezometers are the same as for monitoring wells. Volume of water for sampling may be limiting because of the small borehole and casing diameter unless located in a very productive formation.

#### A.4.3.1.3 Obtaining Samples of Groundwater for Laboratory Analysis

Wells should be allowed to recover to, or near to, their historical static water levels after being purged before obtaining groundwater samples. However, the shorter the time between purging and sampling, the better the sample. In no event should more than 24 hours elapse between purging and sampling. If more than 24 hours has elapsed, the well must be purged again. A well that does not recover sufficiently in 24 hours to provide sufficient volume for a water sample can be considered dry.

Groundwater samples are obtained using pumps or bailers. Certain parameters may require specialized sampling equipment.

When sampling monitoring wells, the pump or bailer should be lowered into the screened interval if its location is known; otherwise, from the middle third of the standing water column. Wells with permanently mounted submersible pumps, such as water supply wells, are to be sampled at the port closest to the well (i.e., furthest upstream) if access to the casing cannot be obtained.

Samples obtained for labile parameters such as volatile organic compounds (VOC), total organic halogens (TOX), or total organic carbon (TOC) require special sampling equipment since exposure to the air will cause loss of the parameter(s) of concern. VOC samples are taken using a special-purpose VOC bailer. Samples for TOC or TOX may be obtained using a bottom-emptying open-top teflon bailer. Sample must be immediately decanted into the sample container, however, and immediately sealed to prevent or minimize loss of analyte.

VOC's in water may contribute to TOC and TOX but will leave the sample and enter air to which the sample is exposed. For this reason, containers for VOC, TOX, and TOC samples must be filled completely and all headspace eliminated. This is accomplished by filling the containers to overflowing so that a convex meniscus rises over the opening of the sample bottle or vial. The lid may then be carefully placed on the container and sealed. After closing the container, it should be checked for any visible bubbles. If bubbles of air are present, discard the sample and refill the container with freshly drawn sample. (Samples for TOX and TOC must be preserved with sulfuric acid. Sample bottles are supplied with the preservative chemicals already added. If presence of headspace requires that a sample be discarded and a new sample taken, fresh preservative chemicals must be added or a new sample container with preservative must be used.)

Samples for VOC, TOC, and TOX may also be obtained using a bladder pump. Sample should be obtained directly from the pump discharge. The bladder pump should be allowed to cycle the number of times sufficient to have purged itself of any residual water present in the system from previous residual recontamination activities.

Samples for nonlabile parameters may be obtained using conventional bailers, bladder pumps, airlift pumps, or submersible pumps. A sufficient volume of sample should be withdrawn to fill all sample containers if bailers are used. Multiple bailer loads should be composited in a clean inert container and then all sample bottles should be filled from the common source. If using a pump, bottles may be filled sequentially.

#### A.4.3.1.4 Preservation of Groundwater Samples

Groundwater samples require various preservatives. For parameters that require the addition of chemical preservatives, the sample containers normally have the proper amount of the appropriate preservatives added to them by the laboratory. Samples for other parameters may need to be cooled in ice until delivered to the laboratory.

A.4.3.1.5 <u>Field Determinations in Conjunction with Groundwater Sampling</u>
Several parameters that are of interest in evaluating groundwater quality are subject to change before the sample can be delivered to a laboratory. Field measurements of these quantities must be obtained immediately upon sampling. Among the values determined in the field are included:

- o depth to water,
- o depth of well,
- o water temperature,
- o air temperature,
- o conductivity,
- o pH,
- o dissolved oxygen, and
- o redox potential.

Depth measurements have been previously addressed in this procedure. Temperature, conductivity, pH, dissolved oxygen, and redox potential are instrumental measurements that are performed on the water sample as soon as it is acquired since these properties are subject to significant changes as soon as the sample is separated from its environment.

Temperature of the water sample is determined using a full-immersion-type mercury or alcohol in glass thermometer. The thermometer would be fully immersed in the sample to insure an accurate reading. The thermometer must bear a current calibration reference. Thermometers are calibrated against a National Bureau of Standards traceable thermometer before being placed in service and semiannually thereafter.

Conductivity is measured using a conductivity meter. The calibration of the conductivity meter must be checked by the sampler using a conductivity standard. The meter must read or be adjusted to read within ±5 percent of the conductivity of the standard. A record of the calibration check and any adjustments to the instruments should be included in the sampling record.

pH is measured with a pH meter employing a glass electrode. pH meters must be field calibrated prior to use using standard buffers. For a pH reading to be valid, it must lie between two calibrated values. Thus, water with a pH of 5.3 would require use of a meter calibrated at pH 4.0 and 7.0. An alkaline water

sample would require calibration at pH 7.0 and 10.0. The range of pH 4.0 to 10.0 will cover virtually all environmental water samples. Buffers with pH values of 4.0, 7.0, and 10.0 are normally supplied with each meter. More extreme buffers (i.e., greater than 10.0 or less than 4.0) are available for samples expected to fall outside this range. These should be requested if measurement of extreme pH values is anticipated. A record of the calibration check and any adjustments made to the instrument should be entered in the sampling record.

Dissolved oxygen is measured with a dissolved-oxygen meter. These meters must be field calibrated using air-saturated water or water-saturated air as the calibration standard. Air temperature and elevation must also be known for this calibration. A written record of the calibration and any instrument settings and adjustments must be entered into the sampling record. Dissolved oxygen can also be determined chemically with portable test kits.

Air temperature is difficult to measure accurately in the field. If possible, use temperature values given from a nearby weather station. If this is not practical or the site presents unique features that would reasonably result in locally atypical temperatures, air temperature can be measured using a calibrated thermometer that has been carefully dried and placed quietly in a shaded location at least three feet off the ground so that reflected heat does not result in an artificially elevated reading. A barrier may even be placed between the ground and the thermometer if reflection of radiant heat is a significant problem (as might be the case, for instance, on a largely barren site).

Redox potential is measured on pH meters equipped with a millivolt readout option using a special redox potential probe. The probe must be calibrated prior to use with a solution of known stable redox potential. Zoebel's solution is customarily used for this purpose and is supplied with the instrument. Temperature of the calibrating solution and of the water being tested must be accurately known for calibration and measurement purposes. A written record of the calibration and any adjustments must be made in the sampling records.

## A.4.3.1.6 <u>Sampling Seeps and Springs</u>

Seeps and springs are also sources of groundwater that may need to be sampled. Seeps are generally very-low-flow sources and water may accumulate in sufficient

quantity to sample only in depressions. As a result, it is usually impractical to sample seeps for VOC, TOC, or TOX constituents. Sampling seeps without contaminating the sample with sediments or other foreign matter is often difficult or impossible. Complete sampling notes about the location and means of acquisition of the sample need to be made at the time of sampling.

Springs are characterized by a significant sustained flow. This makes them much easier to sample than seeps. A spring should be sampled as close to its source as possible, allowing for a minimum of contact time between the sample and the air once it leaves the producing formation. If the flow rate is high enough and a convenient location presents itself, it is sometimes possible to obtain reliable samples for TOC, TOX, and VOC parameters from springs.

## A.4.3.2 Standard Procedures for Sampling Surface Water

Surface water samples may be obtained from flowing sources such as rivers, creeks, streams, runoff channels, or pipe outfalls or from quiescent sources such as ponds, lakes, impoundments, depressions, or puddles. Dimensions of the sampled water body and the information requirements of the project will have a material bearing on the sampling technique.

Surface water samples should be obtained in such a manner that if multipleportion samples are to be taken, the sampling activity will not introduce contaminants in later portions of the sample. Insuring representativeness of the sample is more difficult with surface waters than with groundwaters since groundwater in a properly purged well may be presumed isotropic. This presumption cannot be made for either quiescent or flowing bodies of surface water.

# A.4.3.2.1 Obtaining Samples of Surface Water from Flowing Sources

Streams and rivers have flow characteristics that make certain regions of the flowing body more appropriate for sampling than others. For most purposes, a sample point should be chosen along a straight reach having a fairly regular cross section of flow. If possible, such a point should be just downstream from a significant riffle to promote thorough mixing of streamflow components. Pools should be avoided as sample points if at all possible since significant physical and chemical anisotropy can develop.

When taking samples from a flowing body, the ideal location for maximizing representativeness is midchannel at one third to one half the depth of the channel. This is the region of maximum flow velocity. Many small streams (which provide the bulk of flowing surface water samples) can be sampled using no specialized equipment beyond boots for wading. Bottles should be submerged to the sampling point with the closure in place and the mouth of the container pointing downstream. Remove the closure and allow the bottle to fill. Remove the container from the stream and replace the closure.

Sample containers containing a chemical preservative should not be allowed to fill completely so as to avoid loss of preservative or contamination of the stream. (The exceptions to this are TOC and TOX containers. These bottles must have zero headspace and so must be completely full. The bottle's profile [i.e., wide body and narrow mouth] means that loss of preservative under normal filling circumstances will be minimal.)

Samples for TOC, TOX, and VOC should be allowed to fill completely. Their closures should be held under the stream's surface and allowed to fill with water. The containers should be sealed under water and closures securely tightened before removing the bottle from the stream. This will insure that there is no headspace or bubbles present in the sample.

Under less than ideal conditions (e.g., very low flow rates or a very shallow stream), it may be necessary to obtain a sample in several small increments. A sample scoop may then be used to accumulate the sample volume required in a clean inert container. All sample increments except VOC, TOC, and TOX may then be obtained from the accumulated sample. VOC, TOC, and TOX sample bottles should be filled directly from the scoop immediately. A notation should be made that this expedient was necessary since volatile organic constituents may be lost from the samples. (A sample scoop for shallow surface water may be made by cutting an oblique section from a clean polyethylene sample bottle.)

Larger streams may need to be sampled from a boat or bridge to obtain an optimal sample. For these purposes, a weighted bottle, extended bottle, Kemmerer sampler, or bailers may be employed to take the sample from a defined depth. Samples for VOC analysis must be obtained using a VOC bailer. TOC and TOX

1.1

bottles will not fit into a VOC bailer; therefore, these sample bottles should be filled from the depth-sampling device as soon as it is retrieved from the water.

## A.4.3.2.2 Obtaining Surface Water Samples from Quiescent Sources

Quiescent water bodies such as lakes, ponds, or impoundments can present significant problems of anisotropy. Inflow and outflow characteristics (if present) as well as volume, depth, area, and evidence of stratification must be evaluated to determine the optimal sampling location(s) given the objectives of the project. If characteristics of the discharge from a quiescent body are of interest, then the discharge structure outfall can be sampled as a flowing water body. If the characteristics of the main body of water are desired, samples from a variety of locations or depths may be needed depending upon the size, shape, and presumed degree of anisotropy of the water body. The same procedures used for sampling large flowing bodies from a boat or bridge would be employed for sampling a pond or other quiescent body.

## A.4.3.2.3 Preservation of Surface Water Samples

Surface water samples and groundwater samples are subject to the same preservation requirements, holding times, and allowable materials of construction for containers. See Section A.4.3.1.4 for information on preservation of water samples. Information on holding times and construction materials for containers is found in Chapter A.5.0.

#### A.4.3.2.4 Field Measurements

The same physical and chemical properties that are field determined for groundwater may be required of surface water as well. The same techniques, instruments, calibration, and data-recording requirements apply for surface water as for groundwater.

Field measurements of surface water characteristics may also involve determination of volume or rate of flow. Volume of a quiescent body is determined by taking a sufficient number of soundings or other depth measurements to develop a profile of the bottom of the water body. From this information and the size and shape of the water's surface, an accurate calculation of the volume in the system can be performed. The exact number and location of depth measurements

would be established by the judgement of the sampler or in a site-specific sampling plan.

Measuring or estimating the rate of flow in streams can be approached several ways depending upon accuracy requirements. These methods include the use of the dilution/mass balance method as well as stage-discharge relationships.

In the dilution mass balance method, a concentrated tracer material of known composition is introduced into the flowing water at a location where the tracer will quickly become mixed with the stream flow. After thorough mixing of the tracer with the stream flow, a sample is withdrawn from the stream and the concentrations of the tracer are measured. Common tracers are fluorescent dyes or soluble cations (such as lithium) not commonly found in surface waters. Since all of the tracer found in the sample is presumed to come from the concentrate:

$$(Conc)_C(Flow rate)_C = (Conc)_{Dil}(Flow rate)_{Dil}$$
 (2)

where

C = concentrated tracer and
Dil = stream flow sample.

Units used must be consistent. Stream flows are often calculated in terms of milliliters per minute (an inconvenient unit of measure) that must be converted to more conventional units such as gallons per minute or cubic feet per second.

In the absence of a control structure of precisely known geometry such as a weir or flume (which have precise relationships between stage and discharge), an estimation of discharge can be made by measuring the velocity of flow and determining the flow cross section by measuring the profile of the stream channel. Flow velocity is best measured at about one quarter to one third of its depth rather than at the surface. Surface velocity is significantly less than the average velocity of the stream.

Finally, if the channel has a known slope, a regular cross section, and fairly uniform and known bed and bank material, a stage discharge curve can be constructed utilizing Manning's equation. Flow velocity or flow rate can then be estimated from a measurement of the depth of the stream.

## A.4.3.3 Standard Procedures for Sampling Soil and Soil-Like Materials

These materials include soils, subsoil, earth fill, and similar substances. Soils and soil-like materials are sampled both to determine their own characteristics and to detect the presence, concentration, and extent of contaminants that may be present. Depending on the physical properties of the materials, samples of unconsolidated material can be obtained from the surface down to considerable depth using hand tools. Greater depth (to 20 or 30 feet in some cases) can be achieved using a split-spoon sampler.

Surface samples are obtained using a spade, trowel, or hand-operated augering device. Trowels are limited to sampling approximately the uppermost 6 to 12 inches of soil. Somewhat greater depths are available with a spade; however, it is still essentially a surface-sampling device. Hand-operated augers are effective to depths of six feet or more in good digging conditions. For deeper samples, use of a split-spoon sampler (preferably with a mechanically actuated drop hammer) or excavation of test pits or trenches is required.

# A.4.3.3.1 <u>Sampling Soil with Hand Tools</u>

For shallow samples, convenience and economy often indicate the use of hand tools to obtain samples of near-surface materials. There are a number of considerations affecting representativeness that must be borne in mind when sampling soil with hand tools. Sampling location selection tends to be convenience-driven or expectation-driven when hand tools are used. This can result in a highly biased sample. To prevent this, sample site locations should be decided in advance and changed only if it is essentially impossible to sample a particular location because of ground conditions.

When the hand tools are trowels or spades, the cross section of the sample is also important. The sampled interval must be of a regular and consistent cross section so that all materials are represented in the final sample in the same proportion that they existed in the field. Samples taken with trowel or auger often poorly represent the lower portions of the sampled interval.

When augers are used, the problem of cross section control is largely eliminated since the auger body and the sample removed will have the same cross section.

Specialized augers are available for preserving the integrity of very wet or very sandy materials that might run or leak out of a conventional auger.

Cross contamination can be a major consideration in sampling soils at several different depths using hand tools. Clean surface soil that falls into the sample hole or cut may mask or dilute trace contaminants farther down in the soil column. Conversely, contaminated surface soil may contribute evidence of contamination extending deeper into the soil column than is in fact the case. This can be eliminated by using short lengths of nesting diameters of well casing to prevent materials from higher up the column from sloughing into the horizon being sampled.

## A.4.3.3.2 <u>Sampling Soil with Split-Spoon Samplers and Excavators</u>

Where greater depth is required than can be conveniently reached with hand tools or where physical consistency, mechanical resistance, or soil profile descriptions must be recovered to depth, power-driven sampling machinery must be used. Split spoons are driven into the soil column by repeated standard hammer blows until refusal is reached (100 blows per foot).

When the spoon has been driven its entire length (or refusal has been reached), the spoon is withdrawn and opened. A section of essentially undisturbed soil of uniform cross section is then available for recovery. The sample hole is cleaned out between samples or casing may be driven to prevent vertical mixing.

Test pits or trenches can be excavated to any depth for which adequate equipment is available. Most test pits or trenches are limited to about 15 feet in depth by equipment reach limitations. A test pit or trench leaves a more or less vertical undisturbed exposed face that can be examined in detail for profile descriptions and for sampling. When sampling material from the sides of a test pit, care is required regarding cross section control.

(Test pits in excess of four feet in depth are excavations governed by Occupational Safety and Health Administration regulations. Side slopes must be sloped or shored in accordance with the requirements of 29 CFR 1926 Subpart P.)

#### A.4.3.3.3 <u>Sample Handling Requirements</u>

Soil samples must often be composited, subdivided, or split for analysis by more than one laboratory. Preserving (or achieving) representativeness can be extremely demanding. If several increments must be composited to obtain an average value sample for a location, mixing of increments and reduction of sample volume must be performed in such a way that the resulting analytical sample contains proportionate representation of all increments. For dry granular materials for which volatile compounds are not a concern, a riffle splitter can be used both for mixing the sample increments and then for reducing the sample size to a manageable quantity. If the material is sticky or cohesive, it may be necessary to tumble the contents in a closed jar, tub, or drum to effect adequate mixing. If volatiles are not a concern, sample reduction by successive quartering is effective if flow through a riffle is difficult.

Mixing and reduction of samples for VOC or TOX analysis (because of the presence of naturally occurring organic compounds, TOC is almost never performed on soils) is fairly difficult and compromises in mixing/reduction efficiency and loss of analyte to the air must be made. These compromises must be based on the number and size of the increments, the expected identity and concentration of the volatiles, and the sensitivity of the data required. The mixing container should minimize headspace to that required to permit mixing. The container should be very tightly closed during all operations. Statistical sample reduction by riffling or quartering may not be practical.

When samples need to be split between laboratories or an archive sample needs to be retained, two different approaches are possible. If it is not important to preserve the original sample stratigraphy, split or archive samples should be collected from a homogeneous mixture of the entire field sample. Alternatively, the raw unprocessed samples can be individually split before any operations are performed on them. When this approach is used, the soil sample mass must be divided longitudinally so that uniform cross-sectional representativeness is maintained in both (all) the splits.

## A.4.3.3.4 Sample Containers for Soil Samples

Soil samples are stored for shipment in wide-mouth glass containers with teflonor aluminum-foil-lined closures. Sample size will vary from 40 milliliters (VOC samples) to a liter or more depending on the battery of tests to be performed. If the soil sample will be analyzed for VOC's, the sample container should be filled as full as practicable to minimize headspace and loss of analyte.

Where the procedure has been approved in advance, there is an alternative procedure for handling soil samples for VOC analysis. A quantity of soil, approximately 10 to 25 grams, is introduced quickly into a tared container with a known volume of methanol added. The methanol dissolves any VOC's in the soil and preserves the sample. The headspace is saturated with methanol vapor which suppresses migration into the headspace by other VOC's. The container is sealed with a teflon-lined lid and reweighed. The difference between the final weight and the tare is the sample weight.

A.4.3.4 Standard Procedures for Sampling Low Mechanical Strength Sediments
Low mechanical strength sediments cannot be recovered using conventional coring,
augering, or split-spoon samplers. Special devices with foot valves to prevent
sample loss are required. These techniques will work for sediments of a sufficiently high liquids content that they are mobile and flowable so that gravity
will permit the foot valve to seat when the sampler is withdrawn.

Sediment samplers may be of two basic types; single unit or staged. The single-unit sampler is for sampling layers of sediment or sludge sufficiently thin so that a single increment sample will recover the entire depth of the deposit. In cases where sediments are deep, several single-increment samplers, each with its own foot valve, are stacked one on top of the other. This enables deep layers of sediment to be sampled. Also, the column of samplers can be broken down as the sample is withdrawn, facilitating handling of what can be a different physical arrangement.

Each increment is considered a whole sample and is decanted directly into sample containers. Samples are generally not preserved except by cooling. Because of the high degree of liquid characteristics, these samples can be split, handled, and composited by the same techniques that would be used for liquid samples in accordance with project-specific sampling plans and field observations. A certain amount of vertical cross contamination is unavoidable with these types of materials and samplers.

Where the material is too thick to be sampled by the above technique but is not yet competent enough to be sampled by coring, split spoons, etc., the use of sampling bombs, weighted Kemmerer samplers, or any other means that allows a sample to be recovered from a specified depth should be employed. Field notes should reflect any special sampling problems or novel techniques that were encountered or used.

# A.4.3.5 <u>Standard Procedure for Sampling Ambient Air for Total Suspended Particulates</u>

Air is sampled for total suspended particulates (and for chemical and other determinations on those particulates) using specially designed high-volume (High-Vol) air-sampling equipment. Air is drawn through a filter at approximately 40 cubic feet per minute for a 24-hour period for a sample.

## A.4.3.5.1 Filter Media for Total Suspended Particulates

The normal filtering medium for High-Vol measurement is an eight-inch-by-ten-inch glass fiber filter. To be in conformance with USEPA's standard method, "Reference Method for the Determination of Suspended Particulates in the Atmosphere" (High Volume Method), the filter medium must have the following characteristics:

- o Dimensions of eight inches by ten inches
- o Retention efficiency greater than 99 percent for 0.3-micron particles
- o Absence of pinholes or other mechanical defects
- o pH between six and ten.

The filters are preconditioned in a controlled environment for at least 24 hours before being tared. Conditioning requirements are relative humidity between zero and 50 percent and a temperature between 15°C and 30°C. The conditioning environment needs to be known and recorded.

All filters should be indelibly numbered before being used. Filters can be purchased from the supplier already numbered or numbers can be assigned in the field.

# A.4.3.5.2 Operating Requirements for High-Vol Samplers

The tared filter is carefully placed in the filter holder of the sampler and the filter clamp is attached and tightened. Then the sampler is turned on and allowed to run undisturbed for 24 hours. The roof of the sampler housing must be in place while the sample is being taken.

The sampler must be run at a rate between 39 and 60 cubic feet per minute for the entire 24-hour period. The samplers used by Earth Sciences have an automatic flow controller that maintains a flow of 40 cubic feet per minute regardless of filter loading. Exact time must be determined for start and stop or else an elapsed-time meter must be part of the sampling system. The units used by Earth Sciences have both an elapsed-time meter and automatic start and stop controls that insure that a sample of the proper size is obtained.

A record of the flow through the sampler is provided by a circular chart recorder. The chart for each day's sample needs to be identified by:

- o date,
- o name of technician,
- o sampler identification number, and
- o filter identification number.

The technician responsible for air sampling will also prepare an air sample record. This record will include the following information:

- o Project number
- o Station or sampler number
- o Date
- o Filter number
- o Initial, final, and average flow rates (should be the same for samplers equipped with flow controllers)
- o Initial and final running-time meter readings
- o Total running time
- o Total air volume
- o Temperature range

- o Barometric pressure range
- o Weather conditions
- o Prevailing wind direction and speed

The technician should attach the flow record to the air sample record as part of the document package. At the end of the sampling period, the filter should be carefully removed from its housing, folded "dirt side in," and returned to the laboratory for reweighing.

## A.4.3.6 Standard Procedure for Determining Surface Radioactivity

Building surfaces, the surfaces of equipment, and building facilities such as bench tops, hoods, duct work, and piping that may have come in contact with radioactive material must be surveyed for superficial radioactivity. Surfaces must also sometimes be tested for removable surface radioactivity. Surveys are conducted with appropriate hand-held detection instruments. Removable radiation is determined using a counting instrument.

## A.4.3.6.1 Requirements for Surface Radiation Surveys

Surface radiation must be measured with the appropriate type of instrument. The instrument type must be chosen based on the type of radioactivity that is reasonably expected to be present. A survey instrument for gamma radiation, for instance, will not be effective for this determination of alpha-particle emissions. The instrument's detector must be appropriate to the type of radioactive particle. The instrument must also have a known detection efficiency for the specific radionuclide(s) that is contributing emissions. In some cases, it may be necessary to utilize several different instruments.

Surface radiation limits are given in terms of disintegrations per minute per 100 square centimeters (dpm/ $100 \text{ cm}^2$ ). Instruments for conducting this survey should have a detector surface of this size. This corresponds to a detector diameter of approximately 4.5 inches. Surface radiation readings can be averaged over an area not to exceed one square meter. Objects with a surface area less than one square meter must have its total surface radioactivity averaged.

To conduct a surface radiation survey, divide the surface to be measured into an arbitrary grid of convenient shape. Usually, this is a square grid but this is not necessarily true in all cases. Take at least five regularly spaced readings within each square meter or smaller area if the object being surveyed has less than a square meter of total surface area. Compute the average surface radiation for the area.

# A.4.3.6.2 <u>Documentation of Surface Radiation Surveys</u>

The following information must be recorded for each surface radiation survey:

- o Date
- o Name of sampler
- o Identification of radiation detector used
- o Identification of target radionuclide(s)
- o Calibration record of instrument for the target radionuclide(s)
- o Project number
- o Object or area being surveyed
- o Grid pattern used for survey
- o Location of each measuring station
- o Average value of surface radiation for each area
- o Calculation of average value <u>including detector sensitivity factor for the target radionuclide(s)</u>

It must be possible to find each measuring point at a later date since some measuring points may also have to be sampled for removable radiation. The location of any "hot spots" must also be unambiguously reproducible since the result of the survey may be used to guide later decontamination efforts.

## A.4.3.6.3 Requirements for Removable Radiation Survey

Removable radiation is determined by firmly rubbing a dry filter paper over a

100-square-centimeter area. The filter is then carefully placed in a plastic bag and promptly submitted to a laboratory for counting. To control the area sampled in this fashion, a mask of cardboard or other suitable material is used. A 100-square-centimeter area is cut out of the mask. The filter paper is rubbed over the entire cutout area against the surface to be tested.

The same sort of grid that is used for the surface radiation surveys is also used for removable radiation sampling. In fact, it is usually the same grid if both types of surveys will be performed. For this reason, precise definition of the location of the grid and positions within the grid is important. In the event that removable radiation is above the limit specified in a facility permit or decommissioning plan, the area will have to be cleaned. It is then vitally important that the sampling location can be found again at a later time.

# A.4.3.6.4 Documentation of Removable Radiation Sampling

The following information should be recorded for all removable radiation samples:

- o Project number
- o Date
- o Sampler's name
- o Location, size, and shape of the sampling grid
- o Location of the sample on the grid

This information is best written on the filter before the sample is taken. Be sure to prepare a suitable number of blanks (labeled just as the samples except identified as blanks) to check for ambient or airborne radiation not strictly associated with the surfaces being sampled. One blank should be prepared for every ten removable radiation samples.

#### A.5.0 Custody Procedures and Documentation

# A.5.1 <u>Integral Reagents and Supplies</u>

Relatively few materials will become integral parts of the samples obtained at this site. The materials that will include:

- o air sample filters,
- o nitric acid preservative,
- o sulfuric acid preservative, and

o wipers.

Filters are not altered in any way. Upon receipt from the supplier, they are transferred to a laboratory which conditions them by exposing them to an atmosphere of controlled relative humidity and temperature for at least 24 hours and then numbers and tares them. The numbered tared filters are placed in envelopes and shipped to the field location where they will be used.

Nitric and sulfuric acid preservatives for water samples are dispensed into sample containers by the laboratory in sufficient volume to yield the final acid concentration or pH required by the various methods. The acids used are all reagent grade or better.

Wipers for determination of removable surface radioactivity are commercially available filter papers. They are not altered in any way prior to use.

## A.5.2 <u>Sample Documentation Procedures</u>

All sampling activities will be thoroughly documented.

#### A.5.2.1 Surface Water Samples

or Herman Law 1.

For surface water samples, whether they are from outfalls or streams, a water sample field collection report will be used. One of these will be completed for each collected surface water sample. Particular features that will apply to this project include the following:

- o Sample depth will be designated "surface" for outfalls and the small stream at the south end of the property.
- o Sample depth in the Arkansas River will be measured with a calibrated rope or folding ruler. Depth will be recorded to the nearest inch or tenth of a foot.
- o Subsurface samples will be obtained using a weighted bottle, Kemmerer, or similar device.
- o Sample-point elevations will be determined by survey except in the case of the Arkansas River. River elevation on the day of sampling will be obtained from the gaging station just south of the plant site.
- o Dissolved oxygen will not be determined.

These sample reports will be maintained at the field office with copies sent to the main office when the samples are submitted for analysis.

#### A.5.2.2 Groundwater Samples

Information on well purging and sampling will be recorded on the Well Evacuation/ Water Sampling Report. All relevant portions of the form will be completed during evacuation or sampling. A minimum of three borehole volumes will be evacuated or to dryness, whichever occurs first. The sample must be obtained within 24 hours of the completion of well evacuation.

## A.5.2.3 Soil and Subsurface Materials

Soil, sediment, subsoil, and solid waste materials (i.e., consolidated sludges) that can be sampled using soil sampling equipment such as augers and split-spoon samplers will be documented on a Soil/Solid Material Field Sampling Report. The type of material must be clearly identified since either soils or wastes may be recorded on this form. A separate form must be used for each sample even if more than one sample was obtained from a particular location. For example, a single sample location may be used to obtain a surface soil sample (top six inches) as well as samples from various depths down to split-spoon refusal. Each sample would have its own form.

Sample locations will be staked in the field and clearly identified by sample number at each location. Location of sample points will be established by survey from an established bench mark.

# A.5.2.4 Sludge Materials

Sludge materials sampling is sufficiently variable that a single form has not been developed to record sludge-sampling operations. Sampling details must be recorded in field notes.

Sludge-sampling operations will be conducted from a floating platform or similar conveyance that will provide support for the sampling personnel and equipment. The sampling plan calls for use of a 50-foot grid for picking sample locations. Grid location must be measured from the location of guide ropes on the side of the impoundments or other effective means.

## A.5.2.5 Air Samples

Air sample collection data will be entered on the Air Sample Data Sheet. The circular graph of the air pump chart recorder will be attached to the form. Wind speed and direction will be obtained from the Muskogee airport weather station.

## A.5.2.6 <u>Instrument Monitoring</u>

Where field monitoring instruments are used to gather information about a sample, that information will be entered on the sample collection form or in the field notebook entry for that sample. The instrument used, the person making the measurement, the actual measured value, time, date, and calibration reference must be included for each instrumental measurement. Other relevant information should also be included. This applies only to such measurements as pH, specific conductance, and detection of organic vapors. If more complex field instrument monitoring will be performed such as soil gas measurements and semiquantitative headspace measurements, the specific procedures for these techniques and their associated documentation requirements must be followed.

Instrument surveys of building and ground surfaces for radioactivity will be recorded on a radiation survey form. Collection of radiation wipe samples is also to be documented on this form.

#### A.5.2.7 Other Documentation

Other tasks involved with sampling will also require specific documentation. Routine instrumental air-quality monitoring (distinct from sample material examination) will have its own forms. Test pits and soil profile descriptions will be logged on their own specific forms as will the visual description of split-spoon samples. Monitoring well installation will be documented on Well Installation Detail forms.

## A.5.3 Sample Containers, Preservation, and Transport

Sample containers are provided by the laboratory with the appropriate preservatives already added to the container. Volume and construction material of the sample containers is governed by the type of analysis that will be performed and the number of analyses. Container types for each analyte and the preservatives used are those approved by the USEPA in SW-846 and other guidance documents.

Samples will be transported to the laboratory using common carriers. Samples are placed in insulated coolers, packed in ice to maintain them at 4°C, and shipped for overnight delivery.

## A.5.4 Field Procedures for Sample Custody

## A.5.4.1 Sample Labels

Sample labels will be filled out completely at the time the sample is obtained. The sample number reported on the label must conform in all regards to the sample number on the sampling form and on the chain of custody form.

## A.5.4.2 Chain of Custody Forms

The chain of custody form is filled out at the time a sample is taken. All the relevant information must be entered at this time. Information on the chain of custody form must agree with the sample label and the sampling form.

#### A.5.4.3 Sample Security and Shipment

If personal custody of samples must be terminated for any reason, the samples and their custody forms must be properly secured. They must be locked in a vehicle or structure into which unauthorized entry would be immediately detected. Authorized access mean that access is restricted to the person currently indicated on the custody form as the official custodian.

When samples are shipped by carrier, the custody forms must be sealed inside the cooler that carries the samples listed on that form. The coolers are then sealed with tape and tamper-indicating seals before being submitted for shipment. Upon arrival at the laboratory, a designated person opens the coolers and checks their contents against the custody form.

#### A.5.5 Laboratory Procedures for Sample Custody

Sample custody in the laboratory is under control of the sample traffic coordinator who will receive the samples for the laboratory by signing the chain of custody form. Laboratory sample numbers are then assigned to the samples. Samples that will be subject to internal chain of custody control will then be placed in a secure area. The sample traffic coordinator will issue appropriate amounts of sample to authorized analysts who must sign for the materials and

return any unused portion of the sample. If an analyst must temporarily suspend custody of a sample, it must be returned to the sample traffic coordinator and be signed back into custody. Copies of the sample traffic-tracking records must be submitted to project management periodically for their review.

## A.6.0 Calibration Frequency and Procedures

# A.6.1 <u>Calibration of Field Instruments</u>

All field monitoring instruments will be calibrated at appropriate intervals. Sample collection devices such as High-Vol air samplers and personal sampling pumps will also require calibration. The frequency and procedure for calibration for each instrument or device in use will be presented in the following sections. The following will require calibration at some frequency:

- o pH meters
- o Conductivity meters
- o Oxygen indicators
- o Flammable atmosphere indicators
- o Colorimetric detector tubes
- o Carbon monoxide detectors
- o OVA's
- o Radiation detectors
- o Radiation counter
- o Personal sampling pumps
- o High-Vol air samplers

Some of these will be calibrated by Earth Sciences personnel. Others will be calibrated by the equipment suppliers.

#### A.6.1.1 Calibration of pH Meters

The meter is calibrated against standard buffer solutions of known stable pH. The calibration values should bracket the expected pH of the sample.

#### A.6.1.1.1 Principle of Operation

The pH meter is essentially a millivolt meter with its readout expressed in pH units. The meter responds to the voltage developed between the glass working electrode and a reference electrode. In most field pH meters, the working electrode and the reference electrode are contained in a single probe body but there are still two electrically separate electrodes in the system. The pH of

an aqueous solution is a measure of the concentration of hydronium ions,  $\rm H_3O^+$ . (Actually, it is a measure of hydrogen ion activity but at the low concentrations involved, activity and concentration can be considered equal.) The pH of a solution is the negative of the logarithm (in base 10) of the hydrogen ion concentration in equivalents per liter. Thus, neutral water (pH equals 7) contains  $10^{-7}$  equivalents of hydronium ion per liter.

In any well-behaved electrochemical system, there is a change in the potential between a working electrode sensitive to the ion present and a reference electrode of -57 millivolts for each 10-fold increase in that ion's concentration. The hydronium ion in water is well-behaved over a wide range of concentrations resulting in a wide linear response of electrode potential versus concentration.

The "standardization" control on the pH meter is used to set the response of the meter to the value of a known pH solution. The "slope" control adjusts the meter's circuitry to properly respond to the -57 millivolt-per-decade signal produced by the electrode.

# A.6.1.1.2 Standardization

Almost all pH meters are first standardized on pH 7. This is the middle of the meter's range. Furthermore, all pH meters used by Earth Sciences are built to rotate the slope of the millivolt versus pH readout around the value of 7.

To standardize the pH meter, make sure the electrode cable is attached to the meter and the protective sleeve is removed from the end of the probe. Immerse the probe in fresh pH 7 buffer. Using the "standardize" or "calibrate" control, adjust the meter to read 7.00. NOTE: Field-durable pH electrodes are somewhat slow to respond and stabilize. However, if a stable reading is not obtained after about one minute of immersion in the buffer, the electrode should be cleaned or replaced at the earliest opportunity.

## A.6.1.1.3 Slope Adjustment

After the meter has been adjusted to pH 7, rinse the probe with distilled water and immerse in a buffer of some pH other than 7. Normally this is a pH 4.00 buffer but other values can be as easily used. pH 4 is usually used because most environmental water samples show a pH between 4 and 7.

With the probe in the second buffer, adjust the slope control until the meter reads the pH of the second buffer. Do not use the "standardize" or "calibrate" control to adjust the meter to the value of the second buffer.

When this adjustment has been performed, recheck the meter readout at pH 7. If the electrode is in good condition and the buffer solutions are fresh, the slope adjustment should have had no effect on the pH reading at 7.

# A.6.1.1.4 General Considerations

The acceptance criterion for calibration is  $\pm 0.05$  pH units. If three iterations of the calibration procedure cannot achieve a stable calibration within this criterion, the unit should be taken out of service until the electrode can be replaced or the meter serviced.

The meter is only calibrated between the values of the buffers used in the calibration. Thus, if the calibration is over the range of 4.0 to 7.0, the instrument is <u>not</u> calibrated for samples with a pH greater than 7 or less than 4. For samples outside the original calibrated range, the instrument must be recalibrated with buffers of appropriate pH to bracket the value of the sample. Buffers are available to cover the range from pH 2 to pH 14. Outside of this range, the conceptual significance of pH is suspect. Calibration between 4.0 and 10.0 is adequate for all but the most unusual or highly polluted waters.

#### A.6.1.1.5 <u>Documentation</u>

pH meters are calibrated prior to being issued for fieldwork. The calibration must be confirmed prior to making field measurements and adjustments must be made if necessary. The "pH" block should be checked in the calibration section of the sample field collection report to document the field calibration confirmation.

The calibration of the instrument should be checked periodically throughout the day. Because conditions of use and quality of water are highly variable, no calibration frequency can be specified to cover all cases. Calibration checks should be performed at least once each day that the instrument is used and as often during the day as necessary to maintain confidence in the results.

## A.6.1.2 Calibration of Conductivity Meters

The meter is calibrated against a standard solution of known conductivity. A one-point calibration is all that is required.

#### A.6.1.2.1 Principle of Operation

The conductivity meter is an electrical resistance meter. Resistivity of a solution is measured by the impedance it presents to the passage of an electrical current through it. Resistance (measured in ohms) is mathematically the inverse of conductivity (measured in ohms<sup>-1</sup> or "mhos"). Because the amount of resistance presented by a solution is an extensive property (dependent on the electrical path length), the measured resistance is normalized to a fixed path length making conductivity an intensive property. The unit of measure of this <u>specific</u> conductivity is micromhos per centimeter.

The resistance-measuring cell has a fixed geometry. It is important to maintain this geometry or the measuring device will cease to produce reliable results. The most common problem affecting cell geometry is an accumulation of sludge, sediment, or debris in the narrow bottom portion of the cell. To prevent this, it is important to rinse the cell thoroughly after each use.

Specific conductance is a measure of the amount of dissolved ionic material in the water. The more dissolved salts in the water, the higher the conductivity because of the greater concentration of charge-carrying species. If samples of varying water quality (especially varying dissolved solids content) are to be measured, it is important that the cell be thoroughly rinsed between samples. Prior to calibrating the instrument, the cell should be rinsed with distilled water until a conductivity of less than 10 is achieved.

#### A.6.1.2.2 Standardization

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The conductivity meter is standardized at one known value on one range. Once a single point is accurately fixed, the instrument will be accurate over the entire scale. Since the various ranges available on the meter differ only by the presence of fixed decade resistors, calibration between ranges is not necessary.

To standardize the meter, rinse the cell with distilled water until a constant low reading is obtained. Then place conductivity standard in the cell and empty the cell. Refill with the conductivity standard and read its specific conductance.

The acceptance criterion for standardization is  $\pm 10$  percent of the true value of the standard. If this criterion is not met, empty and refill the cell again. If the acceptance criterion is still not met, remove the plastic plug from the base of the meter and adjust the thumbwheel until the true value is displayed on the meter.

#### A.6.1.2.3 <u>Documentation</u>

Conductivity meters must be calibrated at least once each day that they are used. Calibration should be checked from time to time throughout the day, especially if the instrument is left unused for an extended period or when waters of substantially varying quality are being measured. Calibration is documented on the water sample field collection report. The calibration box should be checked upon successful calibration of the instrument. Because calibration must be traceable to the standard used, enter the identity of the calibration standard in the general remarks. The identity of the standard is the manufacturer and manufacturer's lot number for a purchased conductivity standard. For a conductivity standard prepared by Earth Sciences, the identifier will be a notebook reference which will be on the standard bottle's label. If the calibration had to be adjusted, a notation to that effect should also be made in the general remarks.

## A.6.1.3 <u>Calibration of Oxygen Indicators</u>

Oxygen indicators are used to measure the oxygen content of atmospheres prior to entry into confined spaces or other areas in which there could be a question of oxygen concentration. They are calibrated in clean air immediately prior to use.

# A.6.1.3.1 Principle of Operation

Oxygen meters use an electrochemical cell that reacts to oxygen to produce a signal that is directly proportional to the partial pressure of oxygen in the air. Atmospheric oxygen diffuses into the cell through a permeable membrane where it is reduced on a gold foil electrode and enters the electrolyte solution. The reduced oxygen ion then migrates to a sacrificial lead electrode where it reacts to produce lead oxide. This electrochemical reaction results in the

generation of a voltage difference across the two electrodes that is directly related to the amount of oxygen diffusing into the cell.

The rate at which oxygen enters the cell is determined by the partial pressure of oxygen in the air. While instrument readouts are almost always in percent oxygen, it important to bear in mind that the instrument does not respond to the percentage composition of the air but rather to partial pressure of oxygen. Clean normal air contains 20.8 percent oxygen by volume regardless of atmospheric pressure (and hence partial pressure of oxygen). For this reason, it is extremely important that the oxygen indicator be calibrated at the same atmospheric pressure at which it will be used to measure oxygen content, or erroneous results will be generated.

## A.6.1.3.2 Standardization

In order to calibrate the instrument, it should be turned on and allowed a few seconds to stabilize. Oxygen-detecting cells are always "on," so a warm-up period is not necessary for the detector element. Any normal atmosphere is a suitable calibration gas with a known oxygen content, 20.8 percent by volume. If some reading other than this is generated by the instrument, the calibration potentiometer should be adjusted until the proper reading is obtained. After adjusting the calibration, the instrument should be rechecked to insure that the calibration is stable and that the instrument continues to read out the proper oxygen content.

Some instruments can also be calibrated using a calibration gas that can have an oxygen content other than 20.8 percent. This then provides for a two-point calibration. If a calibration gas other than normal air is used, the instrument should also be tested in normal air to make sure that its response is linear between 20.8 percent and whatever other concentration was used.

The oxygen-detecting cell is always on even when the measuring instrument is turned off. The lead electrode in the cell is sacrificial so the detecting cell has a limited lifetime. After about a year, the instrument calibration will become impossible and/or the calibration may drift significantly. When these conditions start to develop, the detector cell must be replaced.

# A.6.1.3.3 Documentation

A written record of the calibration of the instrument must be made. This is usually entered on the instrument air-monitoring form. If the instrument was calibrated to normal air, this should be explicitly stated. If a synthetic atmosphere was also used, the manufacturer and manufacturer's lot number of the calibration gas should be entered into the calibration record as well as its true concentration and the value measured on the instrument. If any adjustments had to be made to the oxygen indicator, this should be stated in the calibration record. The identity of the instrument (i.e., manufacturer, model number, serial number, and Earth Sciences' inventory number as required to uniquely identify the instrument) should be recorded.

# A.6.1.4 Calibration of Flammable-Atmosphere Indicators

Flammable-atmosphere indicators are used to detect the presence of flammable vapors in the air. The instruments are calibrated against a synthetic atmosphere with a known concentration of flammable gas.

# A.6.1.4.1 Principle of Operation

The flammable-atmosphere indicator is a Wheatstone bridge circuit, one leg of which is a catalytically active cell. Any combustible gases in the atmosphere being tested are oxidized on the detector cell. This oxidation results in a temperature rise which changes the electrical resistance of the cell. Changing the resistance in one leg of a bridge unbalances the circuit, resulting in the passage of current through the meter circuit and causing the needle to deflect.

Combustible-atmosphere indicators respond to the presence of any flammable gas and read out in percent of the lower flammability limit. Flammable-atmosphere indicators respond to the property of combustibility. They provide no information on the true concentration of the gas or its identity.

Different combustible gases will have different response factors for a combustible gas indicator. It is, therefore, important to know what gas was used to calibrate the instrument and what flammable gases are likely to be present in the atmosphere being tested. The manufacturer of the instrument can supply information of specific gas response factors so that instrument readings can be adjusted if necessary.

# A.6.1.4.2 Standardization

The usual calibration gas for combustible-atmosphere indicators is methane in air at a concentration less than the lower flammability limit. The calibration gas is introduced into the instrument's detector cell through a regulator. It is important that the proper regulator be used for the instrument being calibrated. The instrument operates on the rise in temperature of the catalytic converter generated by combustion of flammables. If gas is flowing through the cell at too high a rate, this will effect cooling of the converter and consequently will give too low a reading. If gas flows through the cell too slowly, an inadequate signal may be developed.

Calibration should be performed every day that the instrument is used. For some instruments, such as the MSA Model 2A Explosimeter, the calibration cannot be adjusted. The instrument should read the proper flammability within ±10 percent after response factors have been adjusted for. If this acceptance criterion is not met, the instrument cannot be used. Other devices are adjustable and should be adjusted to read either the true value of the calibration gas or the true value times the relative response factor for the target flammable gas.

For example, suppose the calibration base is 1.45 percent methane in air. This would have a true flammability of 30 percent of the lower explosive limit (LEL) based on a true LEL of 5 percent methane in air. However, if the flammable gas of concern was not methane but gasoline vapors, this value would need to be adjusted for the response factor for gasoline vapors which is 1.67 compared to methane. This would give an equivalent concentration of 50 percent of LEL referenced to gasoline vapor (pentane).

## A.6.1.4.3 Documentation

The calibration information must be recorded in such a way that it can be unambiguously tied to the measurements for which the calibration was performed. Calibration details are usually entered directly on the air-quality monitoring form but may be recorded elsewhere and a reference to the record entered on the monitoring form. The calibration record should indicate the following:

- o Date
- o Identity of instrument

- o Name of operator
- o Concentration of standard gas
- o Identity of standard gas
- o Manufacturer and manufacturer's lot number of the standard gas
- o Instrument reading before adjustment
- o Any adjustments that were made
- o Any response factor adjustments that were made to the true value of the standard.

Any unique or unusual observations or circumstances pertaining to the calibration or the intended use of the instrument should also be noted in the calibration record.

## A.6.1.5 Calibration of Colorimetric Detector Tubes

Colorimetric indicating tubes cannot be calibrated as such. However, the performance of a batch of tubes can be tested against a standard atmosphere to determine their suitability for use.

#### A.6.1.5.1 Principle of Operation

Colorimetric detector tubes are used to determine the presence and concentration of specific chemicals in the atmosphere. A volume of air is drawn through the tube using a pump. A proprietary chemical mixture in the indicating tube will react with the target chemical to produce a color change. The length of the color change is proportional to the concentration of the chemical in the air.

# A.6.1.5.2 Performance Evaluation of Detector Tubes

Detector tubes should be evaluated prior to use. This is accomplished by preparing a test atmosphere and exposing a tube to it. The measured concentration should agree within  $\pm 25$  percent of the true concentration. Make sure that the instructions for performing the test have been read and understood since different tubes have different volumes of air that must be sampled in order to have the scale read properly.

Test atmospheres are prepared in five-gallon plastic buckets. The volume of pure liquid chemical that must be introduced into the bucket to produce the desired concentration can be calculated using the following equation:

$$V(\mu 1) = (ppm)(MW)(.0187)/\rho(24.45)$$
 (3)

where

MW - molecular weight of the chemical and  $\rho$  - its density.

Drill a hole of suitable diameter to admit the detector tube into the lid of a five-gallon bucket. Seal the hole with a strip of duct tape. Dispense the measured amount of liquid onto a watch glass in the bottom of a five-gallon plastic bucket. Immediately cover the bucket and secure the lid. Allow the chemical to evaporate and reach equilibrium concentration in the enclosed volume. Remove the tape seal and insert the detector tube. Sample immediately.

This test needs to be performed once for each manufacturer's lot number of detector tubes received by Earth Sciences for use.

## A.6.1.5.3 <u>Documentation</u>

Documentation of the colorimetric tube performance check is maintained by the health and safety coordinator. The following information is recorded:

- o Manufacturer of detector tube
- o Lot number of detector tube
- o Target compound(s)
- o Volume of pure chemical used to prepare the test atmosphere
- o Manufacturer and manufacturer's lot number of the chemical used to prepare the test atmosphere
- o Concentration of test atmosphere
- o Measured value
- o Date of test

### o Name of person performing test

The health and safety coordinator will make sure that all detector tubes have been evaluated prior to their use in the field.

#### A.6.1.6 Calibration of Carbon Monoxide Detectors

Carbon monoxide is tested for when entering confined spaces and when work is being performed that can be expected to generate carbon monoxide. The instruments are calibrated against a known concentration of carbon monoxide in air.

## A.6.1.6.1 Principle of Operation

Carbon monoxide detectors electrochemically oxidize any carbon monoxide in the sampled air. The oxidation reaction occurs in a polarographic cell. The current produced by the reaction is proportional to the concentration of the gas in the air. Transport of carbon monoxide into the sensor cell is diffusion controlled.

Unlike the oxygen-detector cell in which oxygen spontaneously generates a signal, the carbon monoxide sensor requires an impressed current to operate. Therefore, the instrument will either need time to warm up or else it will have a constant trickle current to the carbon monoxide sensor cell, resulting in a shortened battery life.

#### A.6.1.6.2 Standardization

This instrument should be calibrated each day that it is used. If there is evidence of zero drift or if high concentrations of carbon monoxide (greater than 200 parts per million) are encountered, more frequent calibration will be needed. The carbon monoxide sensor is calibrated using a low-concentration standard gas. The calibration procedure is straightforward. After the instrument has been zeroed in clean air, the gas source is connected to the instrument and calibration gas is introduced through a suitable regulator. After the reading on the meter stabilizes, it is compared with the true value of the standard. If the reported measurement is within  $\pm 10$  percent of the true value, the calibration can be accepted. If this acceptance criterion is not met, the calibration potentiometer should be adjusted until the instrument reads the true value of the test gas.

The carbon monoxide sensor has a limited life. If calibration becomes impossible or instrument drift becomes a problem, the useful life of the cell has expired and replacement is required.

#### A.6.1.6.3 <u>Documentation</u>

The calibration information must be recorded in such a way that it can be unambiguously tied to the measurements for which the calibration was performed. Calibration details are usually entered directly on the air-quality monitoring form but may be recorded elsewhere and a reference to the record entered on the monitoring form. The calibration record should indicate the following:

- o Date
- o Identity of instrument
- o Name of operator
- o Concentration of standard gas
- o Identity of standard gas
- o Manufacturer and manufacturer's lot number of the standard gas
- o Instrument reading before adjustment
- o Any adjustments that were made

Any unique or unusual observations or circumstances pertaining to the calibration or the intended use of the instrument should also be noted in the calibration record.

# A.6.1.7 Calibration of OVA's

Two types of OVA's may be used. One operates on the principle of photoionization; the other on the principle of flame ionization.

## A.6.1.7.1 Principle of Operation

## A.6.1.7.1.1 Photoionization Detectors

Sufficiently energetic photons are capable of ejecting electrons from many organic molecules. These ejected electrons can be collected, amplified, and

turned into an analog or digital signal whose strength is proportional to the concentration of organic molecules in the air.

In a photoionization organic vapor detector, the energetic photons are supplied by an ultraviolet lamp. Lamps can be obtained in three energies, 10.2 eV, 11.7 eV, and 9.5 eV. The strongest signal is produced when the lamp energy and the ionization potential of the target compound are closely matched. For general purposes, the 10.2 eV lamp is most commonly used.

When in use, the sample is drawn by a fan into the light path where ionization takes place. The electrons are collected on a charged grid. The electrical signal is then amplified and read out as an equivalent concentration.

Photoionization detectors have a very wide range of response factors for different chemicals. In general, photoionization detectors will read on the low side, sometimes very low.

The lamp and ion collection chamber tend to become quite dirty when the instrument is used outside. Frequent cleaning of these components is necessary because accumulations of dust or dirt will significantly degrade instrument performance.

#### A.6.1.7.1.2 Flame Ionization Detectors

When organic chemicals are burned in a hydrogen flame, some of the molecules become ionized. These ions are collected, the signal amplified, and read out as a concentration of organic vapors in the air.

The flame ionization organic vapor detector has a tank of hydrogen gas on board that provides fuel for a small flame. The air sample is pumped into the flame chamber where ionization takes place.

Flame ionization devices are much more linear than photoionization devices and exhibit a much smaller variation in response factors. The flame ionization device is capable of detecting virtually all organic compounds with good accuracy.

The flame ionization detectors used by Earth Sciences also have chromatographic capability. The sample can be passed through a chromatographic column that will cause the components of a mixed sample to separate. They are injected sequentially into the hydrogen flame, producing separate concentration values. This enables the constituents to be (tentatively) identified and quantified. Photoionization devices with chromatographic capability are also available.

## A.6.1.7.2 Standardization

# A.6.1.7.2.1 Photoionization Detectors

The 11.7- and 10.2-eV lamps are calibrated using isobutylene. The 9.5-eV lamp is calibrated using benzene. The calibration-gas cylinder is connected to the probe using a piece of rubber tubing. Then the gas is released into the probe through a regulator. The output of the instrument is compared to the true value of the standard. If agreement is not within  $\pm 10$  percent of the true value, the span control is adjusted to make the instrument read out the true value.

# A.6.1.7.2.2 Flame Ionization Detector

The flame ionization detector instruments used by Earth Sciences are calibrated against a known concentration of methane gas and a true zero gas. In addition, agreement from range to range is also checked as part of the calibration procedure.

The range agreement is tested by turning the instrument on. Ignition of the flame is not necessary for this test. Using the calibrate-adjust knob, some arbitrary concentration value is set on the instrument readout with the scale select set on "X1." Usually a value between four and eight is used for range-balance testing. The instrument is then switched to the "X10" scale. The same value should be obtained as that originally set on the "X1" scale. For instance, if the value of 5 were set on the 1 scale, a value of 0.5 should be seen when the scale is changed to the "X10" setting.

With the scale still set on "X10," set a value between 40 and 80 using the calibration adjust on the instrument. Change to the "X100" scale. The same reading should be obtained.

Using a setting of 5 (or 50), suitable agreement between ranges is 4 to 6 (or 40 to 60). If the scales do not balance, consult the instrument manual for the location and identification of the individual scale-adjusting potentiometers. Utilizing a small screwdriver, adjust the appropriate potentiometers to bring the scales into balance with each other.

After the scales have been balanced, turn on the pump and ignite the flame. Connect the cylinder of zero air. After the reading stabilizes, set the instrument to read zero on the "X1" scale. Then change to the "X10" scale and connect the cylinder of 95 parts per million methane. The instrument should read between 90 parts per million and 100 parts per million. If it does not, consult the instrument manual for the location of the calibration adjustment potentiometer. Using a small screwdriver, adjust the instrument readout to the value of the standard.

Other concentrations of calibration gas can be used. An acceptable calibration range will be  $\pm 10$  percent of the true value.

#### A.6.1.7.3 <u>Documentation</u>

Documentation requirements for calibration of the photoionization detector type and the flame ionization detector type of OVA's are the same. The following information needs to be recorded:

- o Date
- o Identity of instrument
- o Name of operator
- o Concentration of standard gas
- o Identity of standard gas
- o Manufacturer and manufacturer's lot number of the standard gas
- o Instrument reading before adjustment
- o Any adjustments that were made
- o Any response factor adjustments that were made to the true value of the standard

This information should be entered in a calibration record book or field notebook as required by project practice. A unique reference to the calibration record should be entered on the air-monitoring form.

## A.6.1.8 Calibration of Radiation Detectors

Earth Sciences does not calibrate radiation detectors. These devices must be calibrated by a specially licensed facility. Calibration takes place every three months or more often if field performance indicates a need for recalibration. A calibration certificate is supplied by the vendor and/or service company. The certificate is maintained in the project files as proof of calibration.

## A.6.1.9 <u>Calibration of Radiation Counters</u>

The same statements apply to counters as to detectors. Earth Sciences is not capable of performing the calibration on these instruments.

## A.6.1.10 Calibration of Personal Sampling Pumps

Personal sampling pumps are used to take bulk air samples for chemical, particulate, or radiochemical analysis. Air is pumped through a collecting medium at a known stable rate for a measured amount of time.

#### A.6.1.10.1 Principle of Operation

Personal sampling pumps are battery-driven devices used to pull ambient air through a sample collecting medium. This may be a filter for particulates, a charcoal or other adsorbent material tube, or an impinger. These pumps feature a steady pumping rate over relatively long periods of time. They do not read out pumping rate directly. They utilize a rotameter with an arbitrary scale that must be calibrated. Calibration is performed every six months.

#### A.6.1.10.2 Standardization

The flow rates are determined using a bubble displacement calibration device. The sampling pump is connected to calibrator using a rubber tube. The pump is then turned on and soap bubbles are allowed to form in the one-liter volumetric bulb. Once the sides of the bulb have been wetted sufficiently to allow a soap bubble to travel the length of the bulb without breaking, the calibration can begin. This is done by measuring the time required for a well-formed bubble to travel between the zero mark and the one-liter mark in the calibration bulb.

This measurement should be made at least twice for at least three different rotameter settings. These times are then converted to flow rates measured as liters per minute. Use these values to plot a calibration curve for each pump.

# A.6.1.10.3 Documentation

Because of the infrequency of the calibration, this information is recorded in a calibrations notebook that is assigned to the sampling pumps. The following information needs to be recorded for the pump calibration:

- o Name of the person performing the calibration
- o Date of the calibration
- o Identity of the pump
- o Identity of the calibrator
- o Rotameter settings
- o Travel times
- o Flow rates
- o Calibration curve

The calibration should be referenced on the air sampling record.

## A.6.1.11 Calibration of High-Vol Samplers

High-Vol air samplers are calibrated against a multiple plate transfer standard that is traceable to a National Bureau of Standards primary reference. The sampler is calibrated upon installation, after any servicing that could affect the flow rate, and every three months. A single point calibration check is performed weekly.

## A.6.1.11.1 Principle of Operation

A High-Vol sampler draws ambient air at a constant rate through a filtering medium that traps any airborne particulates so that they may be weighed to determine the total concentration of particulates in the air. The particulate material can also be analyzed for its chemical, radiological, or mineralogical characteristics.

High-Vol samplers may be equipped with an automatic flow controller. This consists of a heated wire that is placed in the airstream. As the flow begins to diminish because of the filter becoming progressively more loaded with particulates, the temperature of the wire will increase. This temperature

increase produces a signal that results in an increase in the electrical power being fed to the blower motor, which then brings the airflow back up to the setpoint flow rate.

The flow controller must be disabled when performing multipoint calibrations. However, it can be left on when performing single point weekly calibration checks.

#### A.6.1.11.2 Standardization

To perform a multipoint calibration, first disable the flow controller. Then attach the calibration plate on the blower in the place of the filter. A resistance plate should be inserted. Connect the manometer to the calibration plate assembly and turn on the blower.

Allow the blower to run for about five minutes and then read the pressure drop produced inside the calibration plate assembly on the manometer. Using this pressure drop, read the associated flow rate from the transfer standard calibration curve supplied by the manufacturer. Unscrew the cone from the calibration plate assembly and remove the resistance plate. Replace it with another resistance plate, reassemble the calibration plate assembly, and measure the pressure drop with the new resistance plate. The blower may be turned off when changing resistance plates but must be allowed to run awhile afterward before reading the pressure drop. At least three different resistance plate readings must be made for a multipoint calibration.

Flow rates determined from the calibration curve should be compared with the flow rates indicated on the chart recorder assembly. If the indicated flow rate does not agree within  $\pm 10$  percent of the value from the primary standard curve, use a screwdriver to turn the calibration screw on the chart recorder to set it to the true value of the flow.

## A.6.1.11.3 Documentation

The following information needs to be recorded each time a multipoint or single point calibration is performed:

#### o Date of calibration

- o Name of operator
- o Identity of sampler
- o Primary calibration reference of transfer standard
- o Resistance plate(s) used
- o Manometer reading
- o Equivalent volumetric flow rate
- o Reading of flow indicator
- o Any adjustments made to system

If calibration is a result of maintenance or repair operations, the details of the maintenance or repair performed should be noted in the calibration record.

## A.6.2 Calibration of Laboratory Instruments

The calibration frequency and procedural requirements are spelled out in detail in the approved methods published by USEPA. Laboratory equipment and instrumentation is calibrated and documented in accordance with the methods and procedures presented in the following references:

- o U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Office of Solid Waste and Emergency Response, Washington, D.C.
- o U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

In the event that analyses are performed which are not specifically referenced in either of those publications, the instrument(s) used will be calibrated according to the requirements of some other published method or in accordance with good laboratory practices in the event that no accepted published method is available.

#### A.7.0 Analytical Procedures

Samples will be analyzed in accordance with the most recently published versions of laboratory methods published by USEPA. Tables A-1 through A-10 present analytical methods and detection limits which will be utilized during the course of this project. The volumes referenced in Section A.6.2 of Chapter A.6.0 above

will be used for all parameters covered by these manuals. For any analytes not covered by these manuals, some other accepted published procedure will be used.

## A.8.0 Data Reduction, Validation, and Reporting

#### A.8.1 Data Reduction

## A.8.1.1 Reduction of Field Data

Field data will consist of observations and measurements. Measurement information will be subject to several different types of data reduction operations. The principal ones will be:

- o location of surface points from survey information,
- o construction of soil profiles or other descriptions based on physical and instrumental observations on recovered subsurface materials,
- o measurement of well construction for generation of well construction details and for determination of depth of groundwater,
- o delineation of surface or subsurface contamination features, and
- o determination of direction of prevailing wind direction.

Standard surveying mathematical operations will be used to determine site surface locations and elevations. Data reduction for depth measurements involve nothing more difficult than addition and subtraction from a referenced datum determined by survey. All data manipulations will be subject to review and technical checking as part of the data validation process.

# A.8.1.2 Reduction of Laboratory Data

For this project, virtually all data are machine processed. This increases operating efficiency and eliminates a major source of error. Data inputs, including sample numbers, dilution factors, digestion quantities, etc., are subject to verification during the data validation process.

## A.8.2 <u>Data Validation Criteria</u>

## A.8.2.1 Field Data Validation

Field data are inherently difficult to validate since, in most cases, the ultimate source of the data is lost, so there is no "original" against which measurements can be compared. The exception to this is survey data locating permanent stations in the field. Survey data are validated by having a second crew independently determine the location of ten percent of the permanent stations or features that will be used for reference. Examples of such stations and features are building corners, markers on permanent surface features, temporary bench marks, and monitoring wells. Locations should agree within ±6 inches in the horizontal and vertical planes to constitute acceptable validation of the derived locations.

Other field data will be reviewed for internal consistency and completeness. However, no independent validation of the findings will be possible in many respects. Where more than one person was recording field notes on the same activity, those notes will be compared for general agreement.

Field data will be reviewed for acceptance by a technically competent project staff member who is generally familiar with the site operations. Copies of the field notes will be initialed by the reviewer upon their acceptance. Any notes or data not accepted will be so noted. Any questions involving data acceptability will be resolved by project management personnel before the data may be used for the production of calculations, drawings, or report text.

## A.8.2.2 Calculation Procedures and Validation of Calculations

#### A.8.2.2.1 Format

Calculations will be performed on engineering sketch pad paper. The heading information will be filled out in full for all sheets of a calculation. Except for page numbers and the date (if a calculation package is produced over more than one day), all information on each heading for a multipage calculation should be identical.

The following outline should be followed for each calculation package:

- o A short description of the objective of the calculation package.
- o A description of the conceptual approach to achieve the objective.
- o Enumeration of assumptions, values of constants, and complete references for published information. If unusual, nonstandard, or idiosyncratic methods, equations, or values will be employed, their use must be justified.
- o The actual calculation must be shown in full.

## A.8.2.2.2 <u>Documentation</u>

The source of numerical values used in calculations must be identified. If the source is from published material, it should be given its complete bibliographical reference. If this source is a field observation, the date of the field notes and the initials of the observer should be indicated. If calculation data are derived from a map (distances, slopes, areas, locations, etc.) a copy of the map showing points of measurement should be appended to the calculation set or reference should be made to a drawing number that contains the information used in the calculation. Copies of laboratory data reports used in calculations should be included in any calculation packages that make use of laboratory analytical data.

In many cases, a particular value for calculation purposes must be chosen from a generally accepted range of numerical values. When this is done, a statement of the range and the reason for picking the particular value should be given in the calculation package.

Where there are several commonly accepted calculation procedures available to determine a value of interest, the reason for using the one chosen should be stated. If practical to do so, rough indication of equivalency among the methods should be shown. In situations involving the determination of particularly critical parameter values, calculation by two independent methods should be performed, if possible, to increase the level of confidence in the value reported.

All calculations should be written out in full with units as applicable. The stepwise development of a calculation should be shown. The person performing a calculation should consider that the work will be reviewed by someone less familiar with the project task and with the method. Hence, the mathematical argument should be developed fully and in logical order.

### A.8.2.2.3 Checking Calculations

Upon completion of a set of calculations, a clean copy is made of the calculation sheets and all appended material such as maps, charts, nomographs, etc. The originals are filed with the project records and the check copies are submitted to the project manager.

The project manager will have the calculations reviewed and checked to answer the following three concerns:

- o Was an appropriate methodology employed for determination of the parameters of interest?
- o Are the calculations complete in terms of the objective of the task?
- o Are the calculations correctly executed?

The project manager will route the check copies to people able to answer these questions to his satisfaction.

Statements of methodology, values, and procedure should be marked in transparent yellow for concurrence, transparent red for objection, and orange for question. If the reviewer has reservations or objections concerning the answers to the first two concerns, these should be satisfied before further effort is spent checking the calculations themselves. Unless the objection or reservation was trivial or due to unfamiliarity of the reviewer with the subject matter, a notation of the objection or reservation and its resolution may be made to the project file.

After conceptual and completeness checking, calculations are checked for accuracy. Correct calculations are yellowed and incorrect ones are reddened. If the checker cannot determine how a calculation was performed or a value was

arrived at, the item will be marked in orange and the originator will be asked to explain the calculation. If the question can be resolved without revising the calculation, the checker will write "OK" and his initials in ink on the check copy by the item marked in orange.

Completed check copies will be returned to the project manager for review and disposition. This will generally involve correction, revision, filing, or use in subsequent calculations.

# A.8.2.2.4 Corrections to Calculations

Corrections are minor changes in calculations or results that can be made to the original without damaging its legibility or resulting in wholesale changes to text. Corrections are made by lining through the item to be changed and writing the correct entry adjacent to it. This should be done in ink, and the change should be initialed and dated. Nothing is to be erased from the original once it has been submitted for checking.

# A.8.2.2.5 Revisions to Calculations

If a calculation needs to be revised, the originator or other person designated by the project manager will generate a new calculation sheet(s) identified in the title block as a revision to the original calculation. The revision will address in text the purpose of the revision and its scope, including the page numbers of the original and the date of the revision.

The original calculation will be marked as revised at the start of the revision and at the end of the revised portion. The original will be retained even if the entire calculation package is revised. Revisions to an original calculation package must pass through the checking procedure as though they were new documents. If the revision was due to a problem with a concept or completeness, concurrence on the revision as to these matters must be obtained from the project manager or his designee. If the revision was due to numerical operations, only this aspect will need to be checked.

## A.8.2.2.6 Acceptance of Calculations

As each page of an original is checked, it is initialed by the checker, as is the check copy. After corrections to the original have been made, the project

manager will initial the original page in red as final acceptance of the calculation. Original pages which are <u>revised</u> are not initialed; the <u>revision</u> is initialed for acceptance.

After a calculation has been completed, a copy of the entire original plus revisions is placed in the project file. The original and the check copies are filed separately as a project calculation brief. The calculation briefs will be kept in separately bound books or folders for each project. During the active life of the project, custody of the briefs will be the responsibility of the project manager. Once a project has been closed, the calculation briefs are placed in secure storage. Originals and check copies are not to be used as generally circulating materials for subsequent project purposes. The project file copy or other copies made from the accepted calculation brief will be used for this purpose.

# A.8.2.2.7 <u>Computer Calculations</u>

Computer calculations will be performed using only verified programs. Purchased programs will be considered to have been verified by the vendor. Internally produced programs must be verified prior to use in a project.

Computer calculations will be checked by verification of input values. This verification must be performed by some person other than the one who originally input the data. The computer-generated input summary is compared against the input source documents for accuracy of transcription. The checker will mark in yellow all accurate input values on the summary and mark in red any input errors.

In the event of an input error, the computer calculation will be repeated with the error corrected. If the input values are in machine storage, only the erroneous value(s) need be changed and checked on a new input summary sheet. If all input values must be reentered, all input values must be rechecked. Copies of all checked input summaries will be retained together with the result of the final correct computer-generated calculation.

## A.8.2.3 <u>Validation of Drawings and Figures</u>

## A.8.2.3.1 <u>Drawing Numbering System</u>

All drawings, whether or not they will be included in a report to the client, will be uniquely numbered. The drawing number will consist of the project number, task number, size designation, sequence number, and revision number if appropriate.

Standard size designations will be used; i.e., "A" for 8-1/2 by 11, "B" for 11 by 17, "E" for 24 by 36, and "M" for any other size. If a drawing has been revised, the drawing number will be followed by an "R" followed by the number of the revision. Thus, the sixth 8-1/2-by-11 drawing generated during the course of Project Number P0976, Task 04 would be numbered P0976-04-A6. The second revision of the document would be numbered P0976-04-A6-R2.

# A.8.2.3.2 Assignment of Drawing Numbers

A drawing number is assigned upon final approval of the drawing. Drawing numbers within a project and size category will be assigned in order of approval regardless of the order in which they may have been developed or the order in which they may be presented.

A catalog of drawings will be maintained. The catalog will be organized by project number and will include all graphic materials receiving numbers. The catalog will serve as the source for new drawing numbers as well as the record of all drawing numbers previously assigned. The drawing number will include the number of the drawing, its title, and the date of approval.

## A.8.2.3.3 Working Drawings

Drawings not intended for inclusion in reports or otherwise presented to persons outside Earth Sciences are identified as sketches. Examples of sketches are cross sections developed from field data, drawings used to support calculations or to describe relationships among field locations, and drawings used in preliminary design development. Sketches are numbered in the same way as drawings except that the characters "SK" precede the size designation.

Sketch numbers are not included in the catalog. There will be a list of sketches kept in the project file. The assignment of sketch numbers is the responsibility of the originator of the sketch. Sketches are to be maintained after they have been generated. Sketches will be filed with the project records or calculation briefs as appropriate.

### A.8.2.3.4 Approval of Drawings

When the originator has completed a drawing, a copy is produced and submitted to the project manager or his designee for approval. The copy is stamped as a check print and sequentially numbered.

The project manager will review the drawing, marking in yellow the items that are acceptable and circling in red anything on the drawing that is unsatisfactory. The drawing will be returned to the originator for correction of errors or deficiencies, after which a new check print will be generated and submitted to the project manager with all previous check prints. This process is repeated until the drawing is approved and the drawing number is assigned.

# A.8.2.3.5 Revision of Drawings

If a drawing must be altered after approval, the alteration will result in the drawing being identified as a revision. A copy of the drawing with the changes to be made on it will be prepared by, or under the supervision of, the project manager. The project manager must initial the copy showing the revision before any alterations to the approved drawing are made.

Before alterations to the original drawing are made, a clean copy of the drawing will be obtained for a permanent record. This will be identified as a file copy and will be securely stored.

After the drawing has been revised, a check print will be made and submitted to the project manager for approval by the procedure described in Section A.8.2.3.5. Upon approval of the revisions, the drawing number is changed by appending an "R" and the appropriate sequence number. A copy of the revised drawing will then be made and attached to the file copy of the original drawing together with the initialed revision order. Subsequent revisions will be similarly attached to provide a record of all changes made to the drawing.

### A.8.2.3.6 Filing of Drawings

Original drawings on mylar or other reproducible media are stored in flat files with drawers dedicated to specific projects. This is the designated storage area for reproducibles of active project drawings. These files also serve as a repository for hard copies of computer-generated drawings that are not made into reproducibles. A hard copy of <u>all</u> computer-generated graphics will be maintained in the drawing file. For projects utilizing computer graphics, each drawing generated will be backed up and stored on disc(s) dedicated to that project. These project graphics backup discs will be kept in secure storage during the life of the project.

Certain drawings, such as graphs of water quality over time, are meant to be updated periodically. For these drawings, only the most recent edition needs to be kept on file as long as all data from the first and subsequent editions are displayed. Updating such drawings does not constitute a revision. If or when new data eliminate older data, each edition on which this happens will be a revision and copies will need to be retained of each drawing from which data will be removed by updating.

When a project or task has been completed, all drawings are assembled and placed in secure storage. All revision records are similarly stored. Check print sets may be discarded at this time at the project manager's option. Computer backup discs may be moved to secure long-term storage.

# A.8.2.4 <u>Validation of Laboratory Data</u>

#### A.8.2.4.1 Introduction

Before data can be released by the laboratory, the QA/QC team will conduct a review of analytical records for completeness and conformance with stated project requirements. The following sections describe specific review items, acceptance criteria, and remedial actions appropriate to each analyte or group of simultaneously determined analytes.

## A.8.2.4.2 <u>Inorganic Analysis Review and Acceptance</u>

Inorganic analysis covers the following routine laboratory determinations:

- o Metals by flame atomic absorption spectrophotometry (AA), high-temperature graphite atomizer, inductively coupled argon plasma, hydride generation, and colorimetry.
- o Specific anions and cations by colorimetry, automatic analyzer, titration, ion selective electrodes, and gravimetry.
- o Other physicochemical properties by gravimetry, freezing point, titration, colorimetry, and electrometric methods.

## A.8.2.4.2.1 Analytical Instrument Operating Records

Part of the data review involves inspection of instrument operating records. For each instrument or device with a material bearing on data quality, a written or machine-printed record of information supporting the proper functioning and calibration of the instrument or device will be maintained.

## A.8.2.4.2.1.1 pH Meters

All pH meters in use will be uniquely numbered and a calibration record will be maintained. Data approval for pH determinations and potentiometric titrations utilizing pH will involve inspection of the bench data sheet for completion and consistency. The bench data sheet should identify the pH meter used, the date, and the analyst as well as the reported pH of each sample. The calibration book should contain the instrument number, the date, the analyst, and the identity and standard values of the buffers used for the calibration.

Acceptance criteria for pH measurements consist of:

- o complete and internally consistent documentation as stated above,
- o calibration performed on day and shift of analysis,
- o reported values within calibrated range, and
- o calibration within  $\pm 0.05$  units of buffer standard values.

These acceptance criteria apply also to the pH measurement portion of titrimetric determinations.

If project data meet all acceptance criteria, the reviewer will accept the data. Except as specified below, if data do not meet these acceptance criteria, enter a written statement of the deficiency. Upon completion of the review, the bench data sheets and associated project records for any deficient analyses will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the bench data sheet and writing the word "Accept" next to the initials. Acceptable analyses are placed in the project data file.

pH meters are normally calibrated to values between 4.0 and 10.0. Unless a project-specific QA/QC plan calls for calibration outside this range, measured pH values less than 4.0 or greater than 10.0 may be accepted if the instrument had been calibrated to the nearer extreme of the normal range.

pH meters are also used to measure oxidation/reduction potential or Eh. For Eh determination, the following information must be entered in the calibration record:

- o Identification of meter
- o Identification of electrode
- o Eh value of reference standard
- o Identity of reference standard
- o Temperature of standard
- o Measured Eh of standard

Bench sheets recording Eh values will contain the calibration reference for the meter and probe.

## A.8.2.4.2.1.2 Conductivity Meter

All conductivity meters in use will be uniquely numbered and a calibration record will be maintained. Data approval for conductivity measurements will involve inspection of the following documents for completion and consistency. The bench data sheet should identify the conductivity meter used, the date, and the analyst as well as the reported conductivity of each sample. The calibration book should contain the instrument number, the date, the analyst, and the identity and standard value of the conductivity standard used for the calibration.

Acceptance criteria for conductivity measurements consist of:

- o complete and internally consistent documentation as stated above,
- o calibration performed on the day and shift of the analysis, and
- o calibration value within  $\pm 10$  percent of standard value.

If project data meet all acceptance criteria, the reviewer will so indicate on the bench data sheet. If data do not meet these acceptance criteria, the reviewer will enter a written statement of the deficiency. Upon completion of the review, the bench data sheets and associated project records for any deficient analyses will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the bench data sheet and writing the word "Accept" next to the initials. Acceptable analyses are placed in the project data file.

# A.8.2.4.2.1.3 Spectronic 20 and 21 Colorimeters

All Spectronic colorimeters in use will be uniquely numbered. An operation and maintenance log and a calibration log will be maintained. Data approval for analyses using these instruments will involve inspection of the following documents for completeness and consistency. The bench data sheet should identify the instrument, analyst, date, analyte, wave length, sensitivity (Spectronic 21), analytical procedure, and the calibration curve for each analyte and each method of preparation. Calibration log information will include:

- o analyte,
- o identity of calibration standards,
- o date of calibration,
- o initials of analyst,
- o calibration bench data,
- o wavelength,
- o plotted calibration curve,
- o instrument identification, and
- o expiration date.

The operation and maintenance log will have a dated record of any maintenance performed on the instrument including replacement of light sources, cleaning of optics, etc. The operation and maintenance log will also record the results of response linearity checks which are performed monthly and following any maintenance. The following information should be entered:

- o Date of test
- o Initials of analyst
- o Identity of absorbance standards
- o Wavelength(s) checked
- o Absorbance readings
- o Calculations
- o Instrument identification

The operation and maintenance log will also record the usage of each instrument. The following information should be included in the operation and maintenance log each time an instrument is used:

- o Date
- o Analyst
- o Analyte
- o Wavelength
- o Project(s)
- o Instrument identification

Acceptance criteria for Spectronic 20 or 21 measurements consist of the following:

- o Complete and internally consistent documentation as stated above.
- o Calibration is current.
- o Response linearity check is current.
- o Instrument readings are in the calibrated range.

If project data meet all acceptance criteria, the reviewer will so indicate on the bench data sheet. If data do not meet these acceptance criteria, the reviewer will enter a written statement of the deficiency. Upon completion of the review, the bench data sheet and associated project records of any deficient analyses will be submitted to the Vice President of Technical Services or his

designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the bench data sheet and writing the word "Accept" next to the initials. Acceptable analyses are placed in the project data file.

## A.8.2.4.2.1.4 Specific Ion Meter and Probe

All specific ion meters and their associated probes will be uniquely identified. An operation and maintenance log will be maintained. Data approval for analyses using these instruments will involve inspection of the following documents for completeness and consistency. The bench data sheet should identify the instrument, probe, analyst, date, and analyte. The bench sheet should also contain calibration information including the concentration and identification of all standards used and instrument settings and readings. The operation and maintenance log should show the date and initials of anyone performing maintenance on the instruments. The operation and maintenance log should also contain the following information for each occasion a specific ion meter is used:

- o Identity of meter
- o Identity of probe
- o Date
- o Analyst
- o Analyte
- o Project(s)

Acceptance criteria for specific ion meter measurements consist of the following:

- o Complete and internally consistent documentation as stated above.
- o Correlation coefficients of opening and closing curves written within the limits specified in the analytical method.
- o Measurements are within the calibration range.

If project data meet all acceptance criteria, the reviewer will so indicate on the bench data sheet. If data do not meet these acceptance criteria, the reviewer will enter a statement of the deficiency. Upon completion of the review, the bench data sheet and associated project records of any deficient analyses will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the bench data sheet and writing the word "Accept" next to the initials. Acceptable analyses are placed in the project data file.

# A.8.2.4.2.1.5 Automatic Analyzer

Each automatic analyzer and detector module will be uniquely identified. An operation and maintenance log will be maintained. Data approval for analyses using these instruments will involve inspection of the following documents for consistency and completeness. The instrument printout should identify the instrument, analyte, analyst, date of analysis, and calibration information. Calibration information will include the concentration and identification of all standards, instrument readings on these standards, and the calibration correlation coefficient. The operation and maintenance log will show the date and initials of anyone servicing or performing maintenance on the instrument. This log will also contain the following information for each time and instrument is used:

- o Identity of instrument
- o Module configuration
- o Identity of modules
- o Timing program reference
- o Date
- o Analyst
- o Project(s)

Acceptance criteria for automatic analyzer measurements will consist of complete and internally consistent documentation as outlined above. The automatic analyzer will not perform unless preset initial calibration and continuing calibration requirements are met. Any over-range samples must be diluted and rerun for those samples to be acceptable.

If project data meet all acceptance criteria, the reviewer will so indicate on the instrument printout. If data do not meet these acceptance criteria, the reviewer will enter written statement of the deficiency. Upon completion of the review, the printout and associated project records of any deficient analyses will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the printout and writing the word "Accept" next to the initials.

#### A.8.2.4.2.1.6 AA

All AA's will be uniquely identified. Lamps used will also be uniquely identified. An operation and maintenance log and a calibration record will be maintained for the AA's. A record of heating programs is also maintained for the AA equipped with the high-temperature graphite atomizer. Data approval for analyses using these instruments will involve inspection of the following documents for completeness and consistency. The bench data sheet/instrument printout should identify the instrument, lamp, date, analyst, analyte, and the calibration book reference. The bench data sheet portion should also carry the heating program reference for the high-temperature graphite atomizer, if used. The calibration log should identify the standards used in calibration. If the instrument prints out a summary of operating conditions, these conditions should be in accord with the analytical procedure's requirements. The computer calculation sheet heading information should be consistent with that on the bench sheet and/or instrument printout. The calibration data coefficient of correlation should be within the limits specified by the method. The operation and maintenance log will have a dated record of any cleaning, maintenance, or adjustments performed on the instrument. The operation and maintenance log will also record the usage of each The following information should be entered in the operation and maintenance log each time an instrument is used:

- o Date
- o Analyst
- o Analyte
- o Instrument identification
- o Lamp identification
- o Program number (or instrument settings if not a standard program)

## o Project(s)

If project data meet all acceptance criteria, the reviewer will so indicate on the printout/bench data sheet. If data do not meet these acceptance criteria, the reviewer will enter a written statement of the deficiency. Upon completion of the review, the printout/bench data sheet and associated project records of all deficient samples will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the printout/bench data sheet and writing the word "Accept" next to the initials.

## A.8.2.4.2.2 Standard Reagents

Standard reagents are used in titrations as reactants in the quantitative determination of certain parameters. Standard reagents are also used as standards to set or to calibrate instrument responses.

Routine project data approval will require that all standard reagents used are properly reported on data forms or in calibration records. Standards notebook review is not required for routine approval but is performed as part of a project audit.

The reviewer will examine project records to insure that the standards appropriate to the analytical method have been referenced in the proper places in the analytical record. If they have been, he will so signify by checking in red the standard reagent references where they occur in the record. If there is a deficiency in reporting standard reagents used in sample analysis, the reviewer will place an "x" in the appropriate location and will describe the nature of the deficiency. Upon completion of the review, the analytical record and associated project records for deficient standard reagent citations will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If the deficiencies are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the analytical record and writing the word "Accept" next to the initials.

As used in this section, "analytical record" may refer to any of the following:

- o Bench sheets
- o Machine printouts
- o Digestion records
- o Calibration records
- o Sample preparation records

It may also mean any other document that indicates, records, or should record the use of a standard reagent. Accepted analytical records will then be reviewed for QC sample acceptance.

## A.8.2.4.2.3 QC Samples

QC samples are of several sorts. Among them are:

- o field blanks,
- o trip blanks,
- o reagent blanks,
- o method blanks,
- o duplicate samples,
- o spiked samples,
- o standard reference materials, and
- o calibration verification standards.

Of these types, field and trip blanks serve as QC's for field activities and are of no concern to the laboratory for its QC concerns. The other types of QC samples are employed to determine the quality of the laboratory's determinations. Method blanks, duplicates, spikes, standard reference materials, and verification standards are run on a regular basis for all projects. Reagent blanks (as opposed to method blanks) are measured if an apparent interference or contamination shows up in a method blank.

The reviewer will examine project data to insure that each of the required QC samples have been run and that acceptable recoveries were achieved. If these conditions have been met, he will so signify by placing a check mark next to the QC sample result. If there is a deficiency in the QC data, the reviewer will place an "x" next to the deficient entry for the specific parameter(s) and will describe the nature of the deficiency. Upon completion of the review, the analysis records for the deficient analyses will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or

accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the analysis record and writing the word "Accept" next to the initials. Accepted analytical records will be placed in the data files.

## A.8.2.4.2.3.1 Method Blank

A method blank is prepared with each batch of samples. It is subject to the same preparation procedure at the same time using the same lot of reagents. If more than 20 samples are processed at a time, a method blank will be produced with every 20 samples or a fraction thereof. A weight or volume of distilled water or other clean medium is processed as though it were a sample, subject to all steps in the procedure and with all reagents added in proper amount and sequence. The method blank should show no analyte above the specified method reporting level. The reviewer shall determine that the required type and number of method blanks have been prepared and analyzed and that no analyte was detected above the reporting level for the analyses performed, or else the reviewer shall note the deficiency.

# A.8.2.4.2.3.2 <u>Duplicate Sample</u>

One sample per matrix type in each set of analyses (or one sample in 20 or a fraction thereof of each matrix type in each set of analyses) must be performed in duplicate. If duplicate analysis results are less than five times the method reporting level, they must have a relative percent difference within ± the method reporting level. If the results are greater than five times the method reporting level, the relative percent difference must be no more than ±20 percent. The reviewer will ascertain that the required number of duplicates have been analyzed and that the acceptance criteria have been met or else the reviewer will note the deficiency.

#### A.8.2.4.2.3.3 Spiked Samples

One sample per matrix type in each set of analyses (or one sample in 20 or a fraction thereof per matrix type in each set of analyses) will be spiked with each analyte to be determined. Spike recovery must be within the range of 75 to 125 percent unless some other range has been specified in a project-specific QA/QC plan or analytical procedure. The reviewer will ascertain that the

requisite number of spikes have been run and that spike recoveries are in the acceptable range or else the reviewer will note the deficiency.

### A.8.2.4.2.3.4 Standard Reference Materials

Standard reference materials are analyzed at least once per set of analyses or at a rate of one in every 20 samples. Quantitation of standard reference materials for inorganic analytes must be between 80 to 120 percent of the true value unless some other acceptable range has been specified for a particular project. The reviewer will ascertain that the requisite number of standard reference material samples have been run and that recoveries are in the acceptable range or else the reviewer will note the deficiency.

### A.8.2.4.2.3.5 Calibration Verification Standards

A calibration verification standard (a standard not used in the calibration of an instrument and derived from a source independent of the calibration standards) will be run immediately after instrument calibration and after every tenth sample. The calibration verification standard recovery must be between 90 and 110 percent of the true value. The reviewer will ascertain that the requisite number of calibration verification standards have been run and that recoveries are in the acceptable range or else the reviewer will note the deficiency.

# A.8.2.4.2.4 Sample Preparation

Each type of sample analyzed will have undergone some sort of preparation prior to analysis. The project record should contain documentation of the sample preparation(s) used.

The reviewer will ascertain that there is a preparation record for each sample in the project. Preparation information will vary with the type of sample and type of preparation. The preparation record should be completely filled out with all information required supplied. If this requirement has been met, the reviewer will so signify by initialing the preparation record. If there is a deficiency in the QC data, the reviewer will place an "x" by the sample preparation record and describe the nature of the deficiency. Upon completion of the review, the preparation records for any deficient preparations will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient

results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the preparation record and writing the word "Accept" next to the initials. Accepted preparation records will be appropriately filed.

# A.8.2.4.3 Organic Analysis Review and Acceptance

Organic analysis covers the following routine laboratory determinations:

- o TOC
- o TOX
- o Organic compounds in various matrices: by gas chromatograph (GC)/flame ionization detector, by GC/mass spectrometer (MS), by GC/halogen detector, by GC/photoionization detector, and by GC/electron capture detector.

## A.8.2.4.3.1 Analytical Instrument Operating Records

Part of the data review involves inspection of instrument operating records. For each instrument or device with a material bearing on data quality, a written or machine-printed record of information supporting the proper functioning and calibration of the instrument or device will be maintained.

#### A.8.2.4.3.1.1 TOC Analyzer

Each TOC analyzer is uniquely identified. An operating log, calibration record, and instrument parameters record are maintained. Data approval for TOC determinations will involve inspection of the following documents for completion and consistency. The operating log will identify the analyst, date, identification of samples and standards, and the run number. The calibration record will show the date, analyst, and results of the calibration runs. The instrument parameters record will indicate the date and the critical instrument settings.

Acceptance criteria for TOC instrumentation records consist of the following:

- o Complete and internally consistent documentation as described above.
- o Calibrations performed on day and shift of analysis with no instrument off time in between.
- o Reported values within the calibrated range.

If project data meet all acceptance criteria, the reviewer will so indicate on the instrument printouts by entering "instrument OK" and initialing the entry. If data do not meet these acceptance criteria, the reviewer will enter a written explanation of the deficiency on the instrument printout. Upon completion of the review, the printouts and associated project records for deficient analyses will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the printout and writing the word "Accept" next to the initials. Approved records will be submitted for further review of QC data acceptability.

## A.8.2.4.3.1.2 TOX Analyzer

Each TOX analyzer is uniquely identified. An operating log and calibration record are maintained. Data approval for TOX determinations will involve inspection of the following documents for completion and consistency. The operating log will identify the analyst, date, identification of samples and standards, and the run number. The machine-printed record will contain the instrument readings on each sample or standard together with any hand calculations performed.

Acceptance criteria for TOX instrumentation records consist of the following:

- o Complete and internally consistent documentation as described above. Instrument printout must be tied to the operating log.
- o Calibration performed on day and shift of analysis with no instrument off time in between.
- o All reported analyses within the calibrated range.

If project data meet all acceptance criteria, the reviewer will so indicate on the analysis report by entering "instrument OK" and initialing the entry. If data do not meet these acceptance criteria, the reviewer will enter a written explanation of the deficiency on the analysis report. Upon completion of the review, the analysis report and associated project records for any deficient analyses will be submitted to the Vice President of Technical Services or his

designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the analysis report and writing the word "Accept" next to the initials. Accepted analysis reports will be submitted for further review of QC data acceptability.

## A.8.2.4.3.1.3 GC

Each GC is uniquely identified. The following records are kept for each GC:

- o Operating log
- o Calibration record
- o Instrument operating parameters
- o Chromatogram of standards and samples
- o Analysis record

Data approval for GC determinations will consist of a review of the following documents for completeness and consistency. The operating log will identify the instrument, analyst, date, identification of standards and samples, detector in use, column in use, and the run number. The calibration record is a computer-generated report and contains the following information:

- o Analyst
- o Date
- o Column number
- o Detector number and type
- o Instrument condition printout number
- o Analytical method
- o Standards concentration
- o Standards notebook reference or other unique tracer
- o Instrument readings
- o Response factors
- o Average response factor
- o Standard deviation of response factor
- o Correlation coefficient of response factors

The instrument condition printout is a dated machine-printed record of the operating program in use by the instrument. The printout will have a unique number assigned to it composed of the date, instrument identification, and a sequence number. The number on the condition printout and the condition printout number on the calibration record should agree.

The chromatogram should contain the run number, date, time, sample identification, and any dilution factor used. The actual chromatogram of the compound and a printout of the area (in arbitrary units) for each peak should also be presented.

The analysis results report is a computer-generated document. It will contain the following information in addition to the concentration of the analyte:

- o Analyte
- o Analyst
- o Calibration reference
- o Run number
- o Weight/volume of sample
- o Dilution factor
- o Method number
- o Date of analysis

Acceptance criteria for GC data will involve a review of all the above-mentioned documents for internal consistency and completeness. Specifically, the following items must be examined:

- o Agreement between the operating log and calibration record for identification of standards, column number, and detector number and type.
- o Agreement between condition printout number on condition printout and on calibration record.
- o Agreement of run number in log and on calibration and analysis reports.
- o Consistency of dates and names.
- o Calibration acceptance criteria have been met. Unless otherwise stated in the project records, these criteria will be correlation coefficient greater than or equal to 0.95 and relative standard deviation of response factors less than or equal to 20 percent.

Certain analytical protocols will require calibration verification prior to running any samples and a calibration check from time to time thereafter. If required, the documentation for these checks must be in order and the recoveries must be within the required range. These values will be given in the project's supporting documents.

o All analytical results are within the calibrated range.

If project data meet all acceptance criteria, the reviewer will so indicate on the calibration record by entering "instrument OK" and initialing the entry. If data do not meet these acceptance criteria, the reviewer will enter a written explanation of the deficiency on the calibration record. Upon completion of the review, the instrument printouts and associated project records will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the calibration record and writing the word "Accept" next to the initials. Approved records will be submitted for further review of QC data acceptability.

#### A.8.2.4.3.1.4 MS

The MS data package will be the same as that for the GC with the following additions:

- o Tuning standard ion chromatogram will be included in the calibration package.
- o Relative ion abundance calculation sheet will be included in the calibration package.
- o Specific ion chromatogram will be presented for each analyte quantified.

Data acceptance requirements are the same as for GC analyses with the following addition: The results of the tuning standard relative ion abundance check must be within the limits specified for the particular analytical method.

# A.8.2.4.3.2 Standard Reagents

Standard reagents are used in the quantitative determination of certain parameters. Standard reagents are also used as standards to set or to calibrate instrument responses.

Routine project data approval will require that all standard reagents used are properly reported on data forms or in calibration records. Standards notebook review is not required for routine approval.

The reviewer will examine project records to insure that the standards appropriate to the cited analytical method have been referenced in the proper places in the analytical record. If they have been, he will so signify by placing a check mark next to the standard reagent reference and entering "Std. OK" on the document and initialing the entry. If there is a deficiency in reporting standard reagents used in sample analysis, the reviewer will place an "x" at the site of the deficient or absent entry and will describe the nature of the deficiency. Upon completion of the review, the document(s) and associated project records will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the document and writing the word "Accept" next to the initials. Accepted records will be submitted for further review of QC data acceptability.

### A.8.2.4.3.3 QC Samples

QC samples are of several sorts. Among them are:

- o field blanks,
- o trip blanks,
- o reagent blanks,
- o method blanks,
- o duplicate samples,
- o spiked samples,
- o standard reference materials,
- o calibration verification standards, and
- o surrogate standards.

Of these types, field and trip blanks serve as QC's for field activities, cross contamination by sample constituents, and contaminated containers. As such, they are of no concern to the laboratory for its QC concerns. The other types of QC samples are employed to determine the quality of the laboratory's determinations. Method blanks, duplicates, spikes, standard reference materials, and verification standards are run on a regular basis for all projects. Reagent blanks (as opposed to method blanks) are measured if an apparent interference or

contamination shows up in a method blank and when a new lot of a preparation reagent is first used.

The reviewer will examine project data to insure that each of the required QC samples has been run and that acceptable recoveries were achieved. If these conditions have been met, he will so signify by placing a check mark next to the QC sample result on the analysis report. If there is a deficiency in the QC data, the reviewer will place an "x" beside the deficient value and will describe the nature of the deficiency including any absence of required QC sample data. Upon completion of the review, the analysis report and associated project records will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the analysis report and writing the word "Accept" next to the initials.

#### A.8.2.4.3.3.1 Method Blank

A method blank is prepared with each batch of samples. It is subject to the same preparation procedure at the same time using the same lot of reagents. If more than 20 samples are processed at a time, a method blank will be produced with every 20 samples or fraction thereof. A weight or volume of distilled water is processed as though it were a sample, being subject to all steps in the procedure and with all reagents added in proper amount and sequence. The method blank should show no analyte above the specified method reporting level. The reviewer shall determine that the required type and number of method blanks has been prepared and analyzed and that no analyte was detected above the reporting level for the analyses performed.

#### A.8.2.4.3.3.2 <u>Duplicate Sample</u>

One sample per matrix type in each set of analyses (or one sample in 20 or a fraction thereof of each matrix type in each set of analyses) must be performed in duplicate. If duplicate analysis results are less than five times the method reporting level, they must have a relative percent difference within  $\pm$  the method reporting level. If the results are greater than five times the method reporting level, the relative percent difference must be no more than  $\pm$ 20 percent. The

reviewer will ascertain that the required number of duplicates have been analyzed and that the acceptance criteria have been met.

# A.8.2.4.3.3.3 Spiked Samples

One sample per matrix type in each set of analyses (or one sample in 20 or a fraction thereof per matrix type in each set of analyses) will be spiked with certain representative analytes. Spike recovery must be within the range of 75 to 125 percent unless some other range has been specified in a project-specific QA/QC plan. The reviewer will ascertain that the requisite number of spikes have been run and that spike recoveries are in the acceptable range.

## A.8.2.4.3.3.4 Standard Reference Materials

Standard reference materials are analyzed at least once per set of analyses or at a rate of one in every 20 samples. Quantitation of standard reference materials for organic analytes must be between 80 and 120 percent of the true value unless some other value is stated in the project documents or the analytical procedures. The reviewer will ascertain that the requisite number of standard reference material samples have been run and that recoveries are in the acceptable range.

#### A.8.2.4.3.3.5 <u>Calibration Verification Standards</u>

A calibration verification standard (a standard not used in the calibration of an instrument and derived from a source independent of the calibration standards) will be run immediately after instrument calibration and after every tenth sample. The calibration verification standard recovery must be between 90 and 110 percent of the true value. The reviewer will ascertain that the requisite number of calibration verification standards have been run and that recoveries are in the acceptable range.

## A.8.2.4.3.3.6 Surrogate Standards

Surrogate standards specified in an analytical method (GC/MS methods and GC pesticides) will be added in the required amount to <u>all</u> standards, samples, blanks, etc., prior to any preparation, dilution, or extraction. Surrogate standard recovery will be calculated for all samples, standards, blanks, etc. Surrogate recovery must be within the limits specified in the individual method or protocol.

### A.8.2.4.3.4 Sample Preparation

Each type of sample analyzed will have undergone some sort of preparation prior to analysis. The project record should contain documentation of the sample preparation(s) used.

The reviewer will ascertain that there is a preparation record for each sample in the project. Preparation information will vary with the type of sample and type of preparation. The preparation record should be completely filled out with all information required supplied. If this requirement has been met, the reviewer will so signify by entering "Prep. OK" on the preparation record and initialing the entry. If there is a deficiency in the QC data, the reviewer will place an "x" next to the unacceptable, questionable, or missing item and will describe the nature of the deficiency. Upon completion of the review, the checklist and associated project records will be submitted to the Vice President of Technical Services or his designee who will order reanalysis or accept the results with the deficiencies uncorrected. If deficient results are accepted, that acceptance must be indicated by initialing and dating the deficiency notation on the preparation record and writing the word "Accept" next to the initials.

#### A.8.3 <u>Data Reporting Scheme</u>

### A.8.3.1 Field Data Reporting

Field data may be collected by Earth Sciences personnel assigned to field operations and oversight duties. These data are entered on a number of special-purpose forms or in field notebooks as appropriate. At the end of the work day or when a specific task has been completed, field data are turned over to the site supervisor or his designee.

Earth Sciences has produced a number of forms for use in field operations. Wherever possible, these forms should be used. All information items should be addressed on each form used--an item left blank is ambiguous. Field information should be entered directly on the form, not transferred to it at a later time from scrap paper notes, etc. The form should be the true <u>original</u> document. All forms should be executed in ink. Corrections should be lined through once and initialed and the correct entry should be made adjacent. All forms must be

signed and dated. If multiple copies of a form are used, the heading information must be entered on each copy and the entire set must be numbered; e.g., Page 1 of 2.

It is recognized that field conditions can be hard on paper. In the event that a field original is damaged or defaced, it is acceptable to transfer the information to a fair copy for subsequent use. However, the copy must be identified as such, the date the copy was made must be indicated, and the field original must be retained regardless of condition. To prevent further deterioration, it should be placed in an appropriately sized envelope, identified, and stapled to the copy or securely filed as an original document.

# A.8.3.1.1 Field Activity Daily Log

Each Earth Sciences employee involved in a field activity will complete this form each day that field activity is performed. If activities are performed on two (or more) projects on a given day, separate forms will be filled out for each.

The field activity log should serve as an executive summary of tasks performed or observations made. Information should be entered in a form understandable to a third party not directly involved in the project's operations.

### A.8.3.1.2 Sampling Records

Complete information on the location of all environmental and waste samples obtained by Earth Sciences must be obtained. Descriptions of sampled materials should be succinct, accurate, and systematic. If an instrument is used to obtain a numerical value for a parameter, the identity of the instrument must be recorded by its inventory number.

Various types of sampling forms can be used depending on the type of material. The following general guidance should be used in assignment of sample numbers. Every sample identifier should begin with the project number, followed by a sample point designation, followed by a sequence number if appropriate. Descriptive or trivial sample identifiers are to be avoided. Descriptive information should be included on the field sample record but not used as sample numbers.

For example, Monitoring Well No. 8 for Project 5XXX is consistently indicated on project maps as GW8. Therefore, a sample from this well on a given day would be identified as 5XXX-GW8. If more than one sample was obtained from the well on a particular day, they would be numbered further as 5XXX-GW8-1 and 5XXX-GW8-2. If ten samples are obtained from Test Pit 2, identified in project records as TP-2, the samples would be numbered 5XXX-TP-2-1 through 5XXX-TP-2-10.

It is extremely important that sample point location designations be used completely and consistently throughout the life of a project. Sample locations must be indicated on maps permanently in the project files so that analytical data can always be related unambiguously to their physical source.

#### A.8.3.1.3 Chain of Custody

The person obtaining samples has the responsibility for executing the chain of custody record and for making sure that documented custody is unbroken. The chain of custody form must be filled out in its entirety and the person filling out the form must make sure that sample numbers, dates, etc., agree exactly among the sample containers, sampling forms, and chain of custody records.

The chain of custody record will be surrendered to the laboratory upon delivery of the samples. A copy of the countersigned record should be secured for project files.

## A.8.3.1.4 Exploration Records

Various types of intrusive explorations are conducted by Earth Sciences. These include soil profiling, trenching, test pit excavation, and drilling. Each has different methodology and different information is obtained. The following general requirements for documentation should be observed regardless of the type of exploration.

The location of the exploration should be mapped and identified in a consistent manner. Accepted nomenclature and descriptions of rock and earth materials should be used if possible. If idiosyncratic descriptions must be used, they should be clear and concise. The depth or location of significant items should be noted as they are observed. Depths or locations of sampled materials should be indicated.

## A.8.3.1.5 Field Notebooks

The field notebook is the repository of information or observations not directly entered onto the various field activity forms. The notebook also includes data and measurements for which specific forms do not exist.

Each field activity should be noted in the notebook. Each activity should have its own page. In addition to any specific data or observations entered, information on date, time, weather, project and task number, persons present, and planned activities should be recorded. Each page should be signed as it is completed.

The field notebook shall be retained intact by the employee. Relevant pages of the notebook will be photocopied and included in the field activities files. There are no restrictions on information recorded in this field notebook. Any observation that may relate to a project should be recorded so that it may be referred to later.

#### A.8.3.1.6 Field Memoranda

Any item of information, exceptional circumstances, thought, impression, explanation, or observation that bears upon the status of progress of a project and that is not thoroughly addressed in the standard field activity documentation should be written down as a memo to the project manager, site supervisor, team leader, safety officer, etc., as may be appropriate. Information in the memorandum should be accurate and detailed as it will serve to guide the understanding of others. The narrative should be clear and concise. While some information may not be immediately task-related, it should be germane to the project objectives. Memoranda are included in the project documents as part of the permanent record.

Copies of all field records will be maintained at the Earth Sciences field office on the site. Originals will be forwarded to the main office to the attention of the project manager, his designee, or other appropriate person.

Field information will be distributed by the project manager to other project technical personnel for particular tasks such as the production of maps, calculations, and reports. Upon completion of these tasks, the new work product and the field information on which it is based will be returned to the project manager,

who will have formal custody of all in-process information and work products. Upon completion of a task, the work product and associated field information will be placed in the project file.

## A.8.3.2 Laboratory Data Reporting

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After a group of samples has been received by the laboratory and the intake paperwork has been found to be acceptable, the information on the samples including the number of samples, the analytical program, due date, billing information, and other relevant data is forwarded to the Laboratory Information Management System (LIMS) operator who enters this information on the project into computer memory storage. The sample intake data are then placed in the project file.

After an analyst has generated data on the sample, they are submitted to the appropriate group leader for review and approval. Upon approval of analytical data, they are transferred to the LIMS operator who enters the analytical data into the system. The completed analytical data records are then placed in the project file.

Information on sample preparation and instrument operation and maintenance are separately maintained in the analytical group or instrument operating areas. These are not subject to routine review for data approval but are available for inspection at all times.

When all the analytical information has been accumulated for a project, the LIMS operator will prepare a summary of the analytical data. These data are then sent to the word processing department for final report preparation. The final report is then reviewed by a competent laboratory manager for completeness and accuracy before being signed as a final report.

All project-specific information is included in the project file where it can be examined if any questions arise or if an audit needs to be performed. Information in the project file can be used to determine the appropriate preparation and instrument records to examine in the event of an audit.

# A.9.0 Internal Quality Control Checks

## A.9.1 Field QC Checks

Field QC checks will be used to establish the adequacy of sampling procedures and field determinations of contaminant/constituent levels. This is distinguished from field QA procedures which determine the adequacy and completeness of work product documentation, instrument calibrations, and adherence to standard operating procedures.

## A.9.1.1 Sampling QC

Sampling QC will evaluate the ability of field personnel to obtain representative samples of isotropic materials. The only material to be sampled at the site for which the presumption of isotropy can be made is groundwater. Soils, sludges, other subsurface materials, and concentrated waste materials cannot be presumed isotropic such that two independently obtained samples should exhibit the same analytical values of key parameters. For each round of samples obtained in the field, replicate samples will be taken of each groundwater monitoring system; i.e., one replicate from the deep wells and one replicate from the shallow wells. Evaluation of analytical results of these replicates will permit project management to determine whether or not sampling activities are being carried out in such a way that representative samples are being obtained, provided that the laboratory analysis was demonstrably in a state of analytical control. In the event that field replicates do not show a relative percent difference of ±25 percent, sampling procedures will be reviewed and good technique will be reinforced prior to the next round of sampling.

Another aspect of sampling QC is the documentation of the adequacy of decontamination of sampling equipment between locations. Decontamination checks will be provided by the production and laboratory analysis of equipment blanks. One equipment blank will be produced for each distinct matrix sampled and at a rate of 1 equipment blank in 20 samples for each matrix. This requirement will be waived for any materials sampled using disposable sampling devices, or for which decontamination is not required (as with High-Vol air samplers). Failure to recover either analytes known to be present in associated samples or substances used for equipment decontamination will indicate that decontamination has been thorough and acceptably performed. Laboratory recovery of environmental

contaminants in the equipment blank will indicate inadequate equipment decontamination. Recovery of decontamination materials will indicate that field equipment is being inadequately rinsed after decontamination. Either finding will result in review of decontamination procedures and practices and their correction as indicated by the particular findings.

#### A.9.1.2 Measurement QC

Measurement QC will be achieved by challenging instruments used to obtain field measurements with known value materials. Instruments subject to this sort of performance evaluation include the following:

- o Organic vapor analyzers
- o pH meters
- o Conductivity meters
- o Detector tubes
- o Specific gas monitors
- o Combustible gas monitors
- o Radiation detectors

QC checks on monitoring instruments will consist of exposing them to a suitable standard reference material other than the standard(s) used for their calibration. An acceptable determination of the standard's value will constitute a successful QC check. Failure to pass an instrument QC check will result in an evaluation of both the instrument and the operator's technique to isolate the source of the problem and allow for its correction.

#### A.9.2 Laboratory QC

Laboratory QC has been addressed in Chapter A.8.0 in the context of data validation procedures. The different types of QC samples that are routinely run are briefly identified and described below.

# A.9.2.1 <u>Method Blanks</u>

A method blank consists of an aliquot of distilled water or other clean medium which is taken through the entire analytical process, including all preparation steps, exactly as though it were an analytical sample. Method blanks are used to detect reagent contamination and other artifacts that may be introduced by the laboratory. One method blank is prepared per each lot of samples processed at the same time by a single procedure.

# A.9.2.2 Reagent Blanks

Reagent blanks are prepared by combining all the laboratory reagents that are used in a particular analytical procedure in the quantities and in the order that they would be employed. Reagent blanks are used to detect contaminants in reagents that may appear as artifacts in the analysis. One reagent blank is prepared with each lot of a reagent that is used in sample preparation. In the event that a method blank shows evidence of contamination, an additional reagent blank may be prepared to isolate the cause of the contamination observed.

# A.9.2.3 Cleanup Column Blank

If a special cleanup column must be used to prepare complex matrix samples for organic analysis, a solvent blank is passed through the prepared cleanup column to determine that the cleanup column is not itself adding any contaminants to the sample preparation to be purified.

# A.9.2.4 Trip Blank

A trip blank consists of a full set of containers for each type of sample that will be taken in a sampling round. These containers are filled with distilled water at the laboratory and included with the shipment of containers. The trip blank accompanies the field sample containers and field samples at all times. The trip blank is used to detect contamination originating in the containers or due to cross contamination and migration of waste constituents after sampling.

# A.9.2.5 Replicate Sample

A replicate sample is produced in the laboratory by taking a sample of material that is either presumed homogeneous or that can be made homogeneous without risking loss of the analyte of interest and splitting it into two or more aliquots. Each aliquot is then passed through the entire preparation and analysis process. Preparation and analysis of replicate samples checks the reproducibility of laboratory operations. Candidate samples for replicate analysis should be samples that are believed to contain evidence of contamination or to have a measurable concentration of some other species of interest. One replicate sample is produced for every 20 samples of each matrix that is amenable to the process of splitting into aliquots.

# A.9.2.6 Spiked Samples

A spiked sample is an aliquot of a replicate sample that has had a known quantity of a particular analyte or analytes deliberately added to it prior to any sample preparation procedures. Recovery of the spiked analyte(s) is a measure of the laboratory's ability to extract and quantify that particular species from a given matrix. One spiked sample is prepared for every 20 samples of a given matrix from a lot of samples, given that it is of a matrix that can be split without incurring critical and uncontrolled loss of target analyte.

### A.9.2.7 Calibration Standards

Calibration standards are chemical mixtures of known and documented composition and purity. Calibration standards are used to calibrate analytical instrument responses to precisely defined concentrations of target analytes. Instruments are normally calibrated immediately prior to use unless some other frequency of calibration is specified in the instrument's standard operating procedure.

# A.9.2.8 System Performance Check Standards

When organic analysis is performed using GC/MS, system performance check standards are analyzed periodically to evaluate overall performance of the measuring system. System performance check standard recovery is compared to initial calibration values for the same compounds to check for tuning degradation, changes in column performance, etc.

# A.9.2.9 <u>Continuing Calibration Standards</u>

Certain instruments, such as GC and GC/MS, can be run for long periods of time between multipoint calibrations, provided that a continuing calibration standard is run periodically. As long as the compounds in the calibration check standard are properly recovered, the instrument's calibration is assumed to still be in a state of control. Continuing calibration standards are usually the low or middle of the calibrated range of the instrument. These standards must be run at least daily.

# A.9.2.10 Calibration Check Standards

A calibration check standard is a chemical mixture of the same grade and precision as a calibration standard but at a concentration different from, and from a source independent of, the calibration standards. It is run immediately after

the instrument is calibrated to confirm the calibration and may be run periodically thereafter to confirm the stability of the calibration. Calibration check standards also serve to confirm the purity and proper preparation of the calibration standards.

# A.9.2.11 Surrogate Standards

Surrogate standards are chemicals that are generally not found in nature but whose analytical behavior is typical of compounds which are or may be found as contaminants. Surrogate compounds are spiked into samples that will be extracted for organic analysis to provide a continuing check on extraction and analytical efficiency. All samples, standards, and blanks receive the proper amount of the surrogate compounds. The surrogate compounds then provide a constant signature on all analyses to demonstrate the proper functioning of the system.

# A.9.2.12 <u>Internal Standards</u>

Internal standards may be added to samples and calibration standards for organic analysis to provide markers of known response for the quantification of analytes that elute near the same time on the chromatogram. Use of internal standards simplifies the calculation of analyte concentration and makes the analytical system more rugged in the face of drift in instrument performance, since quantification is based on relative peak height or area rather than on absolute peak height or area.

# A.9.2.13 Standard Reference Materials

Standard reference materials are chemical mixtures of known composition that have been subject to interlaboratory quality testing. Standard reference materials have well-documented recovery efficiency. Analytical results should be within the allowable range based on this interlaboratory testing to establish that laboratory performance is acceptable for purposes of comparing data produced by other sources. Standard reference materials are run once for every 20 samples analyzed on a particular instrument. Standard reference materials are primarily used for gaging instrument performance.

# A.10.0 Performance and Systems Audits

# A.10.1 Field Performance Audit

Field performance audits will be performed periodically. The first performance audit will take place before the first round of environmental sampling, at the end of the mobilization and site survey phase of the work. The audit will focus on the status of required programs, documentation of activities, and the presence of equipment and supplies required under the terms of the work plan. Specific items to be audited will include:

- o radiation safety program presence, implementation, and record keeping;
- o employee training requirements and documentation;
- o field activity paperwork status;
- o site health and safety programs other than the radiation safety program;
- o calibration records for all instruments used;
- o QA of field measurements and location of landmarks, features, and sample point locations;
- o presence of sufficient equipment and supplies to support upcoming activities;
- o conformance of activities to date with the requirements of the comprehensive work plan; and
- o compliance of any subcontractors with project plan, procedural, and documentary requirements.

The next field audit will take place during or immediately after the first round of sampling. In addition to the items enumerated above (which will be subject to examination at every audit), the sampling or postsampling audit will specifically address the environmental sampling activity. This audit will include not only an examination of all documentary products relating to sampling activities, but also the direct observation of all types of sampling operations by a knowledgeable observer for actual conformance to standard operating procedures and general requirements of good field sampling practice.

Field operations audits will take place from time to time thereafter during the course of the project. At a minimum, there will be one more audit during the course of the planned field activity phase of the site assessment and a comprehensive audit of field activities at the end of this phase of the project. Other audits will be performed based on the findings of audits already performed, observations of project managers, and at the request of the client.

The results of each field audit will be communicated to project management and to the client at his request. This will consist of a detailed report outlining the following:

- o Scope of the audit.
- o Specific programs and documents examined.
- o Field observations.
- o Identification of areas audited that are in conformance with project plans, standard operating procedures, good operating practice, and/or regulatory requirements.
- o Identification of areas audited that are not in conformance with the above.
- o Specific recommendations for improvement or correction if any deficiencies are detected.

The final audit of field operations will be comprehensive. All phases of the field activities in support of the project goals will be examined and a final report will be prepared presenting findings.

## A.10.2 Laboratory Audits

There will be at least two comprehensive laboratory audits performed in support of this project. The first will take place in advance of any samples being submitted for analysis. A recent laboratory project requiring the same or similar analyses and level of QA and QC will be audited in detail. The audit will examine at least the following:

- o Sample receiving facilities.
- o Sample receiving procedures and documentation.

- o Internal custody controls for samples requiring a high level of security.
- o Sample preparation procedures.
- o Sample preparation documentation.
- o Preparation of required QC samples and materials.
- o Analytical procedures.
- o Documentation of analytical procedures.
- o Adherence to holding time requirements.
- o Documentation of adherence to holding times.
- o Completeness of instrument support documentation including:
  - operating logs;
  - maintenance records;
  - column, detector, lamp, detection module, etc.;
  - usage records;
  - system performance checks;
  - laboratory notebooks;
  - supply inventory records; and
  - analyst qualifications.
- o Completeness of data validation program for project data.
- o Recovery of QC samples.
- o Status of laboratory QC documentation for recent projects other than the specific audited project.
- o Adequacy of laboratory equipment to perform project analytical requirements.

This audit will result in a comprehensive report to project management. The laboratory will also receive a copy of the audit report together with any suggestions for correcting any deficiencies or shortcomings that may be detected in the audit.

A similar comprehensive audit will be performed toward the end of the project to assess data quality for the project as a whole. During the course of the project, there will be a number of more narrowly focused audits. The operational areas subject to audit will be chosen based on the first comprehensive audit and on subsequent observations and experience.

# A.11.0 Preventive Maintenance

Field equipment is inspected each time it leaves the field equipment dispatch office and upon its return. Any needed repairs or replacements are made at that time. Equipment that will be assigned for an extended period to a field operation will be inspected each day the device is used. Maintenance, replacement, or repair of field equipment is performed on an as-needed basis.

All laboratory instruments are under service agreement. Sufficient supplies of spare parts and backup instrumentation are available to maintain operations. Different instruments are under different service and maintenance schedules but all receive appropriate preventive maintenance in accordance with the manufacturer's recommendations.

# A.12.0 Precision and Accuracy Assessment

Several measures of overall data quality are used by the laboratory. Data quality information consists of the results of analysis of QC samples. This information is maintained in computer storage and can be sorted by analytical parameter, analytical method, analyst, and instrument. Standard statistical treatments are performed on the accumulated data to determine accuracy, bias, precision, and tendencies over time.

Accuracy is evaluated by the analysis of QC samples of known values of target constituents. The difference between the average recovery of a known value sample and its true value is used to determine the laboratory, analyst, or instrumental accuracy and bias.

Recovery of replicate samples and matrix spike/matrix spike duplicates provides information on precision of the analyses. Recovery of known value samples also

provides precision information as the same known value material is usually analyzed several different times before it expires. This permits the calculation of the standard deviation of the reported recoveries which measures the precision of the analyses.

As another check for interlaboratory performance, the laboratory takes part in several different laboratory certification programs that are based on the successful analysis of performance evaluation samples. States in which the laboratory is certified based on performance evaluation are Oklahoma, New York, and Pennsylvania, and in Ohio, West Virginia, and Michigan by reciprocity.

The laboratory maintains control charts on several different essential parameters for evaluation of performance. These include recovery of knowns, relative percent difference for replicate samples, surrogate standard recovery, and recovery of spikes. The control chart also plots time trend lines for the parameter being measured to determine if there is any systematic change taking place in analytical performance.

These charts are reviewed monthly or when an analytical problem becomes apparent. This periodic review enables laboratory management to keep regular track of laboratory precision and accuracy.

Completeness of data is determined by project review and acceptance procedures. All required analyses are entered into the LIMS. A project is not released for report preparation until all required analyses have been entered. This insures that projects are completed.

Completeness of laboratory documentation is evaluated as part of the data validation process. Missing documentation requires notification of laboratory management and action on management's part to either accept the data or require reanalysis. Periodic audits are performed to evaluate overall laboratory performance and to serve as a means of correcting any persistent shortcomings.

### A.13.0 Corrective Action

# A.13.1 Field Operations

Corrective action will be taken by the site supervisor, project manager, or health and safety coordinator whenever review of documentation or of operations reveals a deviation from standard operating procedures, good field technical practice, or safe work habits. The corrective action undertaken will depend on the specific nature of the problem. Corrective action will consist of additional training, reinforcement of standard operating procedures, or such other actions as may be necessary to insure conformance with work plan requirements.

# A.13.2 <u>Laboratory Operations</u>

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Corrective action in the laboratory will be triggered by audit results, routine data validation activities, or examination of control charts of critical performance parameters. A succession of three related analyses exceeding the warning limit on the control chart, or two out of five related analyses exceeding the control limit, will result in a formal corrective action investigation to identify the source of the out-of-control condition and to implement appropriate corrective action.

Failure to achieve method-required performance characteristics on any two successive analysis runs will also result in the affected group leader conducting a process review with the analyst(s) and technician(s) involved in the analysis operation.

Failure to pass a performance evaluation sample test set for laboratory certification will also result in performance reviews for the affected analytical groups. The laboratory management and group leaders will review procedures, reagent purity, and laboratory technique to identify the source of the performance shortcomings and to implement suitable measures for their correction.

# A.14.0 Reports to Management

Every audit, field inspection, or laboratory inspection will result in a formal written report to project management. Any corrective action investigations conducted by the laboratory management staff with regard to this project will

also be put in writing and submitted to project management. These reports will detail findings, recommendations, and actions taken (if any). QA reports will be written by the person conducting the audit or performance evaluation, usually the project's designated QA officer or his designee. At the end of the project, a comprehensive project QA audit addressing all significant project activities and operations will be prepared and submitted for project management.

Table A-1
Analytical References
Fansteel Metals
Water, Soil, and Waste Characterization

Parameter	Analytical Reference(1)	Water Detection Level <sup>(2)</sup> (µg/l)	Page 1 of 4 Soil Detection Level(2) (μg/g)
Priority Pollutant Metals (Total): (3)			
Antimony	Method 7040/6010	200	20
Arsenic	Method 7060/7061/6010	1	0.1
Barium	Method 7080/6010	10	100
Beryllium	Method 7090/6010	10	10
Cadmium	Method 7130/6010	10	10
Chromium	Method 7190/6010	10	10
Columbium (Niobium)	DC Plasma	0.05	5
Copper	Method 7210/6010	10	10
Lead	Method 7420/6010	100	10
Mercury	Method 7470(W)/7471(S)	0.5	0.01
Nickel	Method 7520/6010	100	10
Selenium	Method 7740/7741 <sup>(4)</sup>	1	0.1
Silver	Method 7760/6010	10	10
Tantalum	DC Plasma	0.1	10
Thallium	Method 7841	1	0.1
Zinc	Method 7950/6010	10	10

Table A-1 (Continued)

Parameter	Analytical Reference(1)	Water Detection Level $(2)$ $(\mu g/1)$	Page 2 of 4 Soil Detection Level(2) (µg/g)
Priority Pollutant Metals (Dissolved): (3)			
Antimony	Method 7040/6010	200	-
Arsenic	Method 7060/7061/6010	1	-
Barium	Method 7080/6010	10	-
Beryllium	Method 7090/6010	10	-
Cadmium	Method 7130/6010	10	-
Chromium	Method 7190/6010	10	-
Columbium (Niobium)	DC Plasma	0.05	•
Copper	Method 7210/6010	10	-
Lead	Method 7420/6010	100	-
Mercury	Method 7470(W)/7471(S)	0.5	-
Nickel	Method 7520/6010	100	-
Selenium	Method 7740/7741 <sup>(4)</sup>	1	-
Silver	Method 7760/6010	10	-
Tantalum	DC Plasma	0.1	-
Thallium	Method 7841	1	-
Zinc	Method 7950/6010	10	-

Table A-1 (Continued)

Parameter	Analytical Reference <sup>(1)</sup>	Water Detection Level $^{(2)}$ $(\mu g/1)$	Page 3 of 4 Soil Detection Level <sup>(2)</sup> (μg/g)
Priority Pollutant Inorganics:			
Total Cyanide	Method 9012	10	1
Total Phenolics	Method 9066	10	1
Priority Pollutant Organics:			
Volatiles	Method 5030/8260	(see Table 2)	(see Table 2)
Acid Extractables	Method 3510 or 3550/8270	(see Table 4)	(see Table 4)
Base/Neutral Extractables	Method 3510 or 3550/8270	(see Table 3)	(see Table 3)
Pesticides and Polychlorinated Biphenyls	Method 3510 or 3550/8080	(see Table 7)	(see Table 7)
Indicator Parameters:			,
Total Organic Carbon	Method 9060	1,000	20 <sup>(5)</sup>
Total Organic Halogens	Method 9020	10	25
MIBK	Method 8260	50	6.25
Alkalinity	Method 310.1/310.2 <sup>(6)</sup>	2,000 (CaCO <sub>3</sub> )	_(7)
Ammonia	Method 350.1	100	-
Calcium	Method 6010	1,000	100
Chloride	Method 325.2/325.3 <sup>(6)</sup>	500	-
Fluoride	Method 340.2	100	-

Table A-1 (Continued)

			Page 4 of 4
Parameter	Analytical Reference <sup>(1)</sup>	Water Detection Level $(2)$ $(\mu g/1)$	Soil Detection Level <sup>(2)</sup> (µg/g)
Nitrate-Nitrite	Method 353.2 <sup>(6)</sup>	100	-
Sulfate	Method 375.4 <sup>(6)</sup>	1,000	-
Turbidity	Method 180.1 <sup>(6)</sup>	0.01 NTU	-
Gross Alpha	Method 900.0 600/4-80-032	2 pCi/l	0.3 pCi/g
Gross Beta	Method 703 D-03-HASL300(8)	3 pCi/l	0.1 pCi/g

<sup>(1)</sup> Method reference used: U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C.

<sup>(2)</sup> Detection levels (DL) listed are under ideal conditions and sample matrices may cause elevated DL to be reported on contaminated materials. Please note soil units for Tables 2, 3, 4, and 7 are micrograms per kilogram.

<sup>(3)</sup> Digestion Method 3010 is used for aqueous samples and extracts for total metals for analysis by flame atomic absorption. Digestion Method 3020 is used for aqueous samples and extracts for total metals for analysis by furnace atomic absorption spectroscopy. Digestion Method 3050 is used for sediments, sludges, wastes, and soils.

<sup>(4)</sup> Primary method reference.

<sup>(5)</sup> Analysis performed on an ASTM Method A (D 3987-85) extraction.

<sup>(6)</sup>U.S. Environmental Protection Agency, 1983, Methods for Chemical analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

<sup>(7)</sup> Dash denotes not applicable.

<sup>(8)</sup> Standard methods.

# Table A-2 Detection Level Criteria for Volatile Organic Analyses Fansteel Metals Water and Soil Characterization EPA Method 8260

Parameter	Water Detection Level $^{(1)}$ $_{(\mu\mathrm{g}/1)}$	Soil Detection Level $^{(1)}$ $^{(\mu \mathrm{g/kg})}$
Acrolein	250	2,500
Acrylonitrile	250	2,500
Benzene	5	50
Carbon Tetrachloride	5	50
Chlorobenzene	5	50
1,2-Dichloroethane	5	50
1,1,1-Trichloroethane	5	50
1,1-Dichloroethane	5	50
1,1,2-Trichloroethane	5	50
1,1,2,2-Tetrachloroethane	5	50
Chloroethane	10	100
2-Chloroethylvinyl Ether	5	50
Chloroform	5	50
1,1-Dichloroethene	5	50
trans-1,2-Dichloroethene	5	50
1,2-Dichloropropane	5	50
cis-1,3-Dichloropropene	5	50
trans-1,3-Dichloropropene	5	50
Ethylbenzene	. 5	50
Methylene Chloride	5	50
Chloromethane	10	100
Bromomethane	10	100
Bromoform	5	50
Dichlorobromomethane	5	50
Chlorodibromomethane	5	50
Tetrachloroethene	5	50
Toluene	5	50
Trichloroethene	5	50
Vinyl Chloride	10	100
Methyl Isobutyl Ketone	50	6,250

 $<sup>(1)</sup>_{\mbox{Reported}}$  detection levels may be higher due to matrix interferences.

# Table A-3 Detection Level Criteria for Base/Neutral Organic Analyses Fansteel Metals Water and Soil Characterization EPA Method 8270

Davidad	Water Detection Level(1)	Page 1 of 2 Soil Detection Level(1)
Parameter	(μg/l)	(μg/kg)
Acenaphthene	10	1,000
Benzidine	50	2,500
1,2,4-Trichlorobenzene	10	1,000
Hexachlorobenzene	10	1,000
Hexachloroethane	10	1,000
bis(2-Chloroethoxy)methane	10	1,000
2-Chloronaphthalene	10	1,000
1,2-Dichlorobenzene	10	1,000
1,3-Dichlorobenzene	10	1,000
1,4-Dichlorobenzene	10	1,000
3,3-Dichlorobenzidine	50	2,500
2,4-Dinitrotoluene	10	1,000
2,6-Dinitrotoluene	10	1,000
Fluoranthene	10	1,000
4-Chlorophenyl Phenyl Ether	10	1,000
4-Bromophenyl Phenyl Ether	10	1,000
bis(2-Chloroisopropyl)ether	10	1,000
bis(2-Chloroethy1)ether	. 10	1,000
Hexachlorobutadiene	10	1,000
Hexachlorocyclopentadiene	10	1,000
Isophorone	10	1,000
Naphthalene	10	1,000
Nitrobenzene	10	1,000
n-Nitrosodimethylamine	10	1,000
n-Nitrosodiphenylamine	10	1,000
n-Nitrosodi-n-Propylamine	10	1,000
bis(2-Ethylhexyl)phthalate	10	1,000
Butyl Benzyl Phthalate	10	1,000
Di-n-Butyl Phthalate	10	1,000
Di-n-Octyl Phthalate	10	1,000
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Table A-3 (Continued)

	Water Detection Level(1)	Page 2 of 2 Soil Detection Level <sup>(1)</sup>
Parameter	(μg/1)	(μg/kg)
Diethyl Phthalate	10	1,000
Dimethyl Phthalate	. 10	1,000
Benzo(a)Anthracene	10	1,000
Benzo(a)Pyrene	10	1,000
Benzo(b)Fluoranthene	10	1,000
Benzo(k)Fluoranthene	10	1,000
Chrysene	10	1,000
Acenaphthylene	10	1,000
Anthracene	10	1,000
Benzo(g,h,i)Perylene	50	2,500
Fluorene	10	1,000
Phenanthrene	10	1,000
Dibenzo(a,h)Anthracene	. 50	2,500
Indeno(1,2,3-cd)Pyrene	50	2,500
Pyrene	10	1,000
1,2-Diphenylhydrazine	10	1,000

<sup>(1)</sup>Reported detection levels may be higher due to matrix interferences.

# Table A-4 Detection Level Criteria for Acid Extractable Analyses Fansteel Metals Water and Soil Characterization EPA Method 8270

Parameter	Water Detection Level <sup>(1)</sup> (µg/l)	Soil Detection Level $^{(1)}$ $(\mu g/kg)$
2-Chlorophenol	10	1,000
2,4-Dichlorophenol	10	1,000
2,4-Dimethylphenol	10	1,000
4,6-Dinitro-o-cresol	50	2,500
2,4-Dinitrophenol	10	1,000
2-Nitrophenol	50	2,500
4-Nitrophenol	50	2,500
P-Chloro-m-cresol	10	1,000
Pentachlorophenol	50	2,500
Phenol	10	1,000
2,4,6-Trichlorophenol	10	1,000

 $<sup>(1)</sup>_{\mbox{Reported detection levels may be higher due to matrix interferences.}$ 

Table A-5
Analytical References
Target Compound List Parameters
Fansteel Metals
Water and Soil Characterization

Parameter	Reference <sup>(1)</sup>	Water Detection Level $(2)$ $(\mu g/1)$	Page 1 of 2  Soil  Detection Level (2) $(\mu g/g)$
Target Compound List Metals (Total	(3)		
Aluminum	Method 7020/6010	100	10
Antimony	Method 6010	10	10
Arsenic	Method 7060/7061 <sup>(4)</sup>	1	0.1
Barium	Method 7080/6010	10	100
Beryllium	Method 7090/6010	5	10
Cadmium	Method 7130/6010	10	10
Calcium	Method 7140/6010	1,000	100
Chromium	Method 7190/6010	10	10
Cobalt	Method 7200/6010	10	10
Copper	Method 7210/6010	10	10
Iron	Method 7380/6010	100	10
Lead	Method 7420/6010	100	10
Magnesium	Method 7450/6010	1,000	100
Manganese	Method 7460/6010	10	10
Mercury	Method 7470(W)/7471(S)	0.2	0.02
Nickel	Method 7520/6010	10	10
Potassium	Method 7610/6010	1,000	100

Table A-5 (Continued)

Parameter	Reference <sup>(1)</sup>	Water Detection Level $^{(2)}$ $(\mu g/1)$	Page 2 of 2 Soil Detection Level <sup>(2)</sup> (μg/g)
Selenium	Method 7740/7741 <sup>(4)</sup>	1	0.1
Silver	Method 7760/6010	10	10
Sodium	Method 7770/6010	1,000	100
Thallium	Method 7841/6010	1	0.1
Vanadium	Method 7910/6010	10	10
Zinc	Method 7950/6010	10	10
Target Compound List Inorganics:			
Total Cyanide	Method 9012	10	1
Target Compound List:			•
Volatiles	Method 5030/8260	(see Table 8)	(see Table 8)
Semivolatiles	Method 3510 or 3550/8270	(see Table 9)	(see Table 9)
Pesticides and Polychlorinated Biphenyls	Method 3510 or 3550/8080	(see Table 10)	(see Table 10)

<sup>(1)</sup> Method reference used: U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C.

<sup>(2)</sup> Detection levels (DL) listed are under ideal conditions and sample matrices may cause elevated DL to be reported on contaminated materials. Please note soil units for Tables 8, 9, and 10 are micrograms per kilogram.

<sup>(3)</sup> Digestion Method 3010 is used for aqueous samples and extracts for total metals for analysis by flame atomic absorption. Digestion Method 3020 is used for aqueous samples and extracts for total metals for analysis by furnace atomic absorption spectroscopy. Digestion Method 3050 is used for sediments, sludges, wastes, and soils.

<sup>(4)</sup> Primary method reference.

# Table A-6 Practical Quantitation Limits for Target Compound List Fansteel Metals Water and Soil Characterization EPA Method 8260

	Practical Quantitation Limi	
	Water	Low Soil/Sediment
Parameter	(μg/l)	(μg/kg)
Chloromethane	10	10
Bromomethane	10	10
Vinyl Chloride	10	10
Chloroethane	10	10
Methylene Chloride	5	5
Acetone	. 50	50
Carbon Disulfide	5	5
1,1-Dichloroethene	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene (Total)	5	5
Chloroform	5	5
1,2-Dichloroethane	5	5
2-Butanone	50	50
1,1,1-Trichloroethane	5	5
Carbon Tetrachloride	5	5
Vinyl Acetate	10	10
Bromodichloromethane	5	5
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
Trichloroethene	5	5
Dibromochloromethane	5	5
1,1,2-Trichloroethane	5	5
Benzene	5	5
trans-1,3-dichloropropene	5	5
Bromoform	5	5
4-Methyl-2-Pentanone	50	50
2-Hexanone	50	50
Tetrachloroethene	5	5
Toluene	5	5
1,1,2,2-Tetrachloroethane	5	5
Chlorobenzene	5	5
Ethylbenzene	5	5
Styrene	5	5
Xylenes (Total)	5	5

State National Control

# Table A-7 Practical Quantitation Limits for Target Compound List Fansteel Metals Water and Soil Characterization EPA Method 8080

	Practical Quantitation Limits	
	Water	Low Soil/Sediment
Parameter	(μg/1)	(μg/kg)
Alpha-BHC	0.1	8.0
Beta-BHC	0.1	8.0
Delta-BHC	0.1	8.0
Gamma-BHC (Lindane)	0.1	8.0
Heptachlor	0.1	8.0
Aldrin	0.1	8.0
Heptachlor Epoxide	0.1	8.0
Endosulfan I	1.0	8.0
Dieldrin	0.10	16.0
4,4'-DDE	0.10	16.0
Endrin	0.10	16.0
Endosulfan II	0.10	16.0
4,4'-DDD	0.10	16.0
Endosulfan Sulfate	0.10	16.0
4,4'-DDT	0.10	16.0
Methoxychlor	0.1	80.0
Endrin Ketone	1.0	16.0
Chlordane	1.0	80.0
Ioxaphene	1.0	160.0
Aroclor 1016	0.1	80.0
Aroclor 1221	0.1	80.0
Aroclor 1232	0.1	80.0
Aroclor 1242	0.1	80.0
Aroclor 1248	0.1	80.0
Aroclor 1254	1.0	160.0
Aroclor 1260	1.0	160.0

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# Table A-8 Detection Level Criteria for Toxicity Characteristic Leachate Procedure Metals Fansteel Metals Solid and Semisolid Characterization

Parameter	Analytical Reference (1)	Detection Level
pH	A; Method 9045	±0.05
Percent Solids	B; Method 160.3	0.1%
TCLP Extraction	A; Method 1311	_(2)
Arsenic (extraction concentration)	A; Method 6010	0.1 mg/l
Barium (extraction concentration)	A; Method 7080/6010	10 mg/1
Cadmium (extraction concentration)	A; Method 7130/6010	0.1  mg/l
Chromium (extraction concentration)	A; Method 7190/6010	0.1 mg/l
Lead (extraction concentration)	A; Method 7420/6010	0.1  mg/l
Nickel (extraction concentration)	A; Method 7520/6010	1 mg/l
Mercury (extraction concentration)	A; Method 7470/6010	0.05 mg/l
Selenium (extraction concentration)	A; Method 7770/7741/6010 <sup>(3)</sup>	0.1  mg/l
Silver (extraction concentration)	A; Method 7760/6010	0.1 mg/l

# (1) References used:

- A = U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C.
- B = U.S. Environmental Protection Agency, 1985, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.
- (2) Dash denotes not applicable.
- (3) Primary method reference.

# Table A-9 Analytical References Fansteel Metals Air Monitoring (Particulate) Characterization

Parameter	Reference(1)	Detection Levels
Hot Extraction	A; S-790	
Arsenic (extraction concentration)	B; Method 7061	0.1 μg
Cadmium (extraction concentration)	B; Method 7131	$0.1~\mu g$
Chromium (extraction concentration)	B; Method 7191	0.1 μg
Lead (extraction concentration)	B; Method 7241	$0.1~\mu \mathrm{g}$
Nickel (extraction concentration)	B; Method 7520	$0.1~\mu g$
Tantalum	C; DC Plasma	10 μg
Columbium (niobium)	C; DC Plasma	5 μg
Fluoride	B; 340.2	0.1 μg
Gross Alpha	900.0 600/4-80-032	0.3 pCi
Gross Beta	703 D-03-HASL300	0.1 pCi

# (1) References used:

- A = Ambient standards; Appendix G Reference Method; The Determination of Lead in Suspended Particulate Matter Collected from Ambient Air; federal regulations; published by the Bureau of National Affairs, Inc., Washington, D.C. 20037, July 31, 1987.
- B = U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C.
- C = Manufacturer's method.

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# Table A-10 Analytical References Fansteel Metals Water, Soil, and Waste Characterization

Parameter	Analytical Reference <sup>(1)</sup>	Water Detection Level <sup>(2)</sup> (mg/l)
TCLP Metals (Solids/Semisolids):		
Arsenic	Method 7060/7061/6010	0.1
Barium	Method 7080/6010	10
Cadmium	Method 7130/6010	0.1
Chromium	Method 7190/6010	0.1
Lead	Method 7420/6010	0.1
Mercury	Method 7470(W)/7471(S)	0.05
Selenium	Method 7740/7741 <sup>(3)</sup>	0.1
Silver	Method 7760/6010	0.1

<sup>(1)</sup> Method reference used: U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C.

<sup>(2)</sup> Detection levels (DL) listed are under ideal conditions and sample matrices may cause elevated DL to be reported on contaminated materials.

<sup>(3)</sup> Primary method reference.

Appendix B

Health and Safety Plan

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Appendix B
Health and Safety Work Plan
Remedial Assessment
Fansteel Metals
Muskogee, Oklahoma

# B.1.0 Introduction

This site Health and Safety Plan (HSP) has been prepared in accordance with the requirements of Occupational Safety and Health Administration Regulation 29 CFR 1910.120(b)(4). The HSP addresses the risks to employee health and safety that may be present at the Fansteel Metals (Fansteel) site and the measures that will be taken to measure and control those risks.

# B.2.0 Preliminary Evaluation

# B.2.1 Location and Size of the Site

The Fansteel site is located near the town of Muskogee, Oklahoma between Highway 165 and the west bank of the Arkansas River. The property consists of 110 acres of which approximately 40 are involved in the manufacturing processes carried out at the facility. The location of the site is shown in Figure 1. The layout of the site and the area to be addressed by the HSP is delineated in Figure 2.

# B.2.2 Task Description

This HSP addresses the conduct of a remedial assessment to determine the characteristics of the contents and the volume of materials in several impoundments and the potential impact of the impoundments on the surrounding environment. Another purpose of the remedial assessment will be to determine the nature and extent of contamination resulting from the failure of a liner in one of the ponds. In order to accomplish these goals, the following tasks will be performed:

- o Install monitoring wells.
- o Obtain soil samples from a variety of depths.
- o Sample the contents of the impoundments.
- o Determine the depth of materials in the impoundments.

- o Sample sediment and surface water runoff.
- o Sample the soil and/or bedrock underlying the impoundments.
- o Conduct subsurface intrusive investigation for presence of buried drums.
- o Conduct geophysical surveys for presence of buried drums or other containers.
- o Obtain wipe samples from process equipment, laboratory areas, and other locations that may have been contaminated by radioactive materials.

Completion of these tasks and subsequent data analysis and evaluation should result in generation of a site remediation plan. Activities which may be conducted in accordance with any such plan are <u>not</u> addressed by this HSP which covers only the tasks conducted for the remedial assessment portion of the work.

### B.2.3 <u>Duration of Tasks</u>

Field activities to be conducted in performing the remedial assessment are anticipated to require approximately eight weeks. Adverse weather conditions or unanticipated difficulties in sampling subsurface materials may result in a longer duration.

#### B.2.4 Site Topography and Access

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The Fansteel site is essentially flat, sloping gently from west to east toward the Arkansas River. There is an intermittent drainage course south of the plant. The major surface features consist of the plant buildings and nine surface impoundments. The site lies approximately 15 feet above the normal pool elevation of the Webbers Falls Reservoir on the Arkansas River.

Access to the site is gained from State Route 165 just north of its intersection with U.S. Route 62. Railroad service is provided by a spur from the Texas and Pacific Railroad. Commercial air service is available through Tulsa, Oklahoma. There are charter and general aviation services available in Muskogee.

# B.2.5 Anticipated Health and Safety Hazards

The activities to be conducted during the remedial assessment will subject employees and employees of contractors to a variety of physical and chemical hazards. Any or all of the following may be expected to be present during all or part of the project activities:

- o Work in proximity of mobile-powered equipment.
- o Work in proximity to impoundments of mechanically unstable materials that may be unable to support the weight of persons or equipment.
- o Work in or around flowing water.
- o Exposure to radionuclides.
- o Exposure to hazardous chemicals and/or hazardous wastes.

Risk analyses and control strategies for these anticipated hazards will be addressed in a following section of the HSP.

# B.2.6 <u>Dispersion Pathways for Contaminants</u>

There are three primary dispersion pathways for the contaminants known or believed to be present: migration to groundwater, overland flow with runoff, and airborne dispersion as dusts. Which pathway will predominate will be governed by prevailing weather conditions, subsurface conditions, and the chemical and geochemical characteristics of the contaminants. Significant vapor-phase migration is not anticipated since the contaminants are for the most part aqueous liquids or solids.

### B.2.7 Emergency Services

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Emergency services would be available through the client, Fansteel, who maintains an on-site medical capability. If ambulance or hospital service, police, fire, or other emergency response is needed, these services are available through the city of Muskogee. Addresses, telephone numbers, and local alerting procedures will be obtained and posted prior to beginning work at the site.

# B.2.8 <u>Hazardous Substances Known or Believed to Be Present</u>

Plant operations are well defined. The following substances are known to be present in various waste streams generated at the facility; no other contaminants are anticipated:

- o Hydrofluoric acid
- o Fluorspar (calcium fluoride)
- o Calcium oxide
- o Calcium hydroxide
- o Sodium metal
- o Methyl isobutyl ketone (MIBK)
- o Ammonium salts
- o Tantalum and compounds
- o Niobium (or columbium) and compounds
- o Uranium
- o Thorium

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The hazardous properties of these chemicals will be addressed in detail in following sections of this HSP.

# B.2.9 Considerations for Initial Entry

An "initial entry" will not be performed. The site has been in operation in excess of 30 years. While refining operations have been recently shut down, the site is still occupied and the risks of the area and the location of probable concentrations of hazardous waste are well known. Rubber gloves and boots will be worn to protect against skin contact with contaminants. If dry and dusty conditions are present, respiratory protection consisting of a particle filter of suitable porosity will be used (MSA Type S or equivalent). A radiation detector capable of responding to alpha-particle radiation will be carried when working with wastes, contaminated materials, or potentially contaminated materials.

# B.3.0 Site Health and Safety Staff

The following personnel will have administrative and operational responsibilities for activities covered by this HSP:

o Alan F. Bilzi - Executive Vice President of Earth Sciences Consultants, Inc. (Earth Sciences).

- o Scott C. Blauvelt (Director of Geosciences) Project Director, Earth Sciences.
- o Joseph M. Harrick Project Manager, Earth Sciences.
- o M. David Tourdot Project Manager, Life Sciences Group, Earth Sciences.
- o Paul N. Taylor Health and Safety Coordinator, Earth Sciences.
- o To Be Named Site Operations Supervisor.
- o To Be Named Site Health and Safety Officer.

At least one person with operational responsibility for the implementation of this plan will be on the site whenever Earth Sciences personnel are performing tasks governed by this plan. At least one person with administrative responsibility for health and safety will be on site or available on call whenever Earth Sciences personnel are performing project-related activities at the site.

# B.4.0 Project Task Risk Assessments

In the following materials, individual project tasks will be described. The risks associated with each task will be identified, and the strategy for controlling or eliminating the exposure to such hazards will be identified and discussed in brief.

# B.4.1 Sampling Surface Soils and Soil-Like Materials

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In several areas (see Figure 2), soils and other materials may have been contaminated by wastes from the facility. These materials include natural in-place soil, fill, gravel, embankments, and old dewatered sludge. These materials will be sampled to determine the presence, concentration, and extent of contamination.

Surface and shallow samples may be taken using a hand auger. This is a low risk, low-hazard exposure activity. The hazards of this operation include the following:

o Exposure to low levels of organic contaminants, primarily if not exclusively MIBK.

- o Exposure to low levels of heavy metals contaminants.
- o Exposure to airborne radioactivity.
- o Exposure to hazardous chemicals used in decontamination of sampling equipment between sampling locations.

Of these, the fourth is probably the most serious hazard.

Appropriate personal protective equipment (PPE) will be worn when sampling soil or other materials that may be contaminated with hazardous waste constituents. This will consist of nitrile gloves and rubber boots unless site observations and instrumental measurements indicate otherwise. If dusty conditions prevail, a suitable particle respirator will be worn.

During <u>any</u> intrusive activity, even surface soil sampling, suitable monitoring instruments will be used. These will consist of an organic vapor analyzer (OVA) of either the photoionization (PID) or flame ionization (FID) detector type capable of responding to MIBK at an ambient concentration of 10 parts per million or lower. Use of a 10.2 eV probe will be satisfactory since the ionization potential of MIBK is less than this value. A radiation detector capable of responding to alpha-particle radiation will also be used to check for locally elevated concentrations of radionuclides.

# B.4.2 Sampling Subsurface Soils and Soil-Like Materials

Samples from depths not conducive to hand auger sampling will also need to be obtained from a number of locations. These will generally be obtained by means of split-spoon sampling. The same chemical exposure risks that were mentioned for surface-soil sampling will apply to this sampling activity as well. Concentrations of organic vapors may be greater for samples farther from a free-air surface. In addition, split-spoon sampling involves working in proximity to mobile-powered machinery.

Suitable caution should be exercised around drilling or spoon-driving equipment. The same type of protective equipment and monitoring devices specified in Section 4.1 will be used when obtaining split-spoon samples. Organic vapor and radiation measurements will be obtained while the spoon is being driven. As soon as the spoon is opened, the length of recovered material will be scanned first

for organic vapor contamination and then for radioactivity. High levels of either, as well as visual cues indicating the presence of contaminants, should be carefully recorded to serve as guidance for health and safety requirements should the materials have to be removed as part of a subsequent project.

# B.4.3 <u>Sampling Unconsolidated Pond Contents</u>

Except for Ponds Nos. 1N, 1S, and 3, all the ponds at the facility contain large quantities of solid or semisolid process residues. Pond No. 3 which received metals processing wastes consisting of liquid and solid materials has leaked through a hole in the liner and has been dewatered through a combination of uncontrolled release and deliberate pumping and treating of the liquid phase. A large but unknown volume of semisolid residues remains in Pond No. 3. Ponds Nos. 1N and 1S are currently used for surge capacity at the wastewater treatment plant. The remaining ponds at the site are full of wastewater treatment residues, principally lime-neutralized hydrofluoric acid. The resulting material is primarily insoluble calcium fluoride, CaF<sub>2</sub>, and excess hydrated lime. The material in these ponds is approximately 40 percent solids.

The material in Pond No. 3 is toxic and corrosive. It is known to contain the following chemical substances:

- o Hydrofluoric acid and fluoride compounds
- o MIBK
- o Residual niobium and tantalum
- o Uranium and thorium

These materials are present in much higher concentrations than might be found in contaminated environmental materials.

# B.4.3.1 Hydrofluoric Acid, HF, and Fluorides

The liquid phase remaining in the pond has been reported to be as high as 10-molar HF. At these concentrations, HF is corrosive to organic tissues and many metals and is capable of destroying glass and other silicate materials. HF is also toxic by ingestion and inhalation. The current permissible exposure level (PEL) for HF is three parts per million. Inhalation of HF vapors can result in severe pulmonary edema and ulceration of the upper respiratory tract at concentrations as low as 50 parts per million. For airborne inorganic

fluorides, the PEL is 2.5 milligrams per cubic meter. It is considered highly toxic by ingestion with an  $\rm LD_{50}$  of less than 50 milligrams per kilogram. Ingestion of fluoride compounds whether orally or by absorption from inhalation causes bone abnormalities and calcification of connective tissues.

## B.4.3.2 <u>MIBK</u>

MIBK was used as an extracting agent in the separation of tantalum from niobium in the refining process. Substantial quantities of this organic solvent were discharged to Pond 3. MIBK has a PEL of 50 parts per million, and is considered slightly toxic (LD $_{50}$  greater than 500 milligrams per kilogram). The principal hazard of MIBK when present in quantity is its low flash point, 62.6°F. Locally flammable conditions could develop on handling this material during hot weather.

# B.4.3.3 Niobium and Tantalum Compounds

Niobium is a ubiquitous element and is found in quantities of approximately 100 milligrams throughout the body. It is a moderate eye irritant and severe skin irritant. Upon ingestion or inhalation, it has been found to damage liver and kidneys in experimental animals. This type of damage is typical of many heavy metals. Tantalum has been linked to some skin injury.

# B.4.3.4 Uranium and Thorium

Uranium and thorium are present in niobium and tantalum ores as significant impurities. They become concentrated in the tailings from the destruction of the ore in the HF digestion process. The material in Pond 3 is estimated to be approximately 0.2 percent in both uranium and thorium.

Uranium and thorium are both radioactive elements. The parent radionuclides are alpha-particle emitters. Daughter products are both alpha and beta emitters. Some gamma radiation is also associated with each of these nuclear transformations. However, the natural radionuclides are not intense gamma emitters. The primary radiation hazard from uranium and thorium or their compounds is carcinogenicity following inhalation or ingestion. Soluble uranium compounds are also acutely toxic although thorium compounds are not.

B.4.3.5 <u>Health and Safety Risk Analysis for Pond No. 3 Sampling Activities</u>
Obtaining a set of representative samples of the remaining material in Pond No. 3 will expose employees to several potentially hazardous conditions. These include the following:

- o Corrosive material
- o Inhalation hazards
- o Flammable atmospheres
- o Engulfment

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o Radioactive materials

Obtaining representative samples will involve personnel having to move over the surface of the remaining semisolid material in Pond No. 3. The mechanical and weight-bearing properties of this material are unknown but suspect. The solid residue is also corrosive. Some sort of secure work platform that can be moved about or above the surface of Pond No. 3 will have to be constructed. Such a platform must have sufficient size and bearing capacity to hold the sampling equipment and personnel needed to obtain the samples. It must be constructed out of materials that will not be corroded by the contents of the pond. Guardrails and other protective devices required under 29 CFR 1910 Subpart D must be provided. A high level of dermal protection and rapid extraction/emergency decontamination system must be provided for all personnel. Appropriate respiratory protection equipment based on organic vapor levels observed at the time of the activity must also be provided.

# B.4.3.6 <u>Health and Safety Risk Analysis for Sampling Water Treatment Residue</u> Ponds

The wastewater treatment ponds are full of lime-neutralized hydrofluoric acid wastewater sludge. The principal ingredients are calcium chloride and excess lime. The material is believed to be quite alkaline. If the pH is controlled by the solubility of calcium hydroxide, the pH should be 12.5. This is not a corrosive material. However, all inorganic fluorides are toxic by ingestion and inhalation.

The most serious hazard known to be facing personnel sampling this material involves its mechanical stability which is unknown and suspect. The ponds present an engulfment hazard. The same sort of work platform required for sampling Pond No. 3 will be required for the wastewater treatment sludge ponds.

The wastewater treatment sludge has not shown elevated radioactivity in previous analyses conducted by the client. However, we cannot know for certain that radionuclides and toxic substances are not present in lower strata of the pond sludge which is as much as 20 feet deep in some areas.

Personnel will require a high level of dermal protection when working on sampling these ponds and respiratory protection as indicated by organic vapor monitoring conducted before and during sampling. Emergency extraction and decontamination facilities must be provided while this material is being sampled. A suitable number of support personnel will have to be available to effect rescue of the sampling crew at all times when they are on the surface of the ponds.

### B.4.4 Installing and Sampling Monitoring Wells

The groundwater is not believed to be heavily contaminated at the site. Groundwater monitoring systems will be installed to <u>detect</u> traces of contamination. Installation and sampling of monitoring wells is a low-hazard activity. The primary hazard involves working around mobile-powered equipment. Chemical and radiological hazards are not anticipated. Minimal dermal protection will be required unless unexpected developments occur. No respiratory protection is anticipated for these operations. Environmental monitoring will be conducted as described in subsequent sections of this plan.

#### B.4.5 Sampling Surface Water and Sediment

The hazards involved in sampling surface water and sediments are essentially similar to those involved in sampling surface soil. The same types of precautions will apply.

## B.4.6 <u>Intrusive Exploration for Buried Drums</u>

One area of the plant (see Figure 2) may have had drums of unknown materials buried in it. This area is too close to major sources of electromagnetic interference to permit reliance on nonintrusive means of exploration. Therefore, a series of test pits or trenches will have to be excavated in this area to test for the presence of buried drums, other containers, concentrations of waste materials, and contamination of soil and other subsurface materials by waste constituents. The following hazards may apply to this activity:

- o Exposure to unknown concentrated toxic substances, possibly HF, MIBK, process residues, or ores.
- o Exposure to contaminated soils or subsurface materials.
- o Exposure to elevated levels of radioactivity, including airborne radioactivity.
- o Presence of flammable liquids or flammable atmospheres.
- o Oxygen deficiency.
- o Work around mobile-powered equipment.
- o Engulfment by pit or trench collapse.

If the test pits or excavations must be entered for any reason, this activity will constitute a confined-space entry and will be subject to all the requirements for conducting such operations.

Exposure to these hazards will be mitigated by use of a ground observer to carefully examine the progress of the excavation for any sign of concentrated wastes, changes in soil color, conformation, or moisture content, and the presence of metallic objects. This person must be in a high level of respiratory and dermal protection against the possibility of encountering unknown concentrated waste materials. Respiratory protection must also be available to the operator of the excavator. Suitable monitoring instruments will be used during the excavation operations. Additional monitoring instruments will be used in the event that the excavation must be entered.

If excavation entry must be performed, the test pit or trench will be constructed in conformance with the requirements of 29 CFR 1926 Subpart P. A "competent person" as defined in the applicable regulations will inspect the excavation prior to entry and shall certify that the excavation is properly constructed and safe to enter. An effective means of extraction will be provided for any persons entering an excavation regardless of anticipated duration of the entry activity.

# B.4.7 Surface Geophysical Exploration for Buried Drums

The area designated Pond No. 5 in Figure 2 may have been used for the burial of drums of HF in addition to surface disposal of process residues. This areas is sufficiently removed from known sources of electromagnetic interference to permit use of an electromagnetic survey to detect buried metallic items. The hazard level for this activity will be minimal. The primary potential risk is from exposure to elevated levels of radioactivity due to the presence of process waste at this location.

Protection of employees involved in this operation will be minimal. Rubber boots should be worn. If dusty conditions prevail, a suitable dust respirator will be provided for protection against airborne radioactivity. Radiation detection instruments will be used to measure occupational exposure to radionuclides.

If buried items are detected, an intrusive exploration will be conducted. This exploration will be subject to the same types of hazards and will employ the same abatement strategy as outlined in Section 4.6 above.

## B.4.8 Obtaining Wipe Samples

Wipe samples will be obtained from building surfaces, process equipment, laboratory equipment, and other plant facilities. These samples will be used to detect the presence of removable radioactivity. Obtaining wipe samples will possibly involve a number of different hazards depending on the location of the surface and the materials that may have been used in the same area. Among the hazards that may be present are the following:

- o Physical hazards involving working at elevated heights.
- o Presence of toxic or corrosive chemical residues.
- o Elevated levels of radioactivity including airborne radioactivity.
- o Confined-space entry.

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Employee protection will be provided by use of suitable dermal and respiratory protection, provision of acceptable work surfaces, and provision of environmental monitoring. Each area or device to be sampled will be examined by the site

safety officer before the activity begins so that proper protective equipment and work practices may be specified.

## B.5.0 Training Requirements

There will be one task activity that will require specific-hazard training in addition to the general site worker 40-hour health and safety training before work can commence. This involves pond sampling operations. Employees who will be involved in this activity will be trained in the following operations:

- o Characteristics of pond contents to the extent known.
- o Operation of sampling platform positioning system.
- o Regulations affecting work on the platform.
- o Safe work practices for sampling sludge from the platform.
- o Communication with support team.
- o Operation of extraction/rescue devices.
- o Emergency decontamination.

A record of this training will be made and maintained on the site. Periodic safety meetings to discuss upcoming activities will be held on a regular basis.

# B.6.0 Personal Protective Equipment

Various tasks identified in Section 4.0 will require some degree of PPE. This equipment and the conditions for its use are described below.

B.6.1 <u>Self-Contained Breathing Apparatus or Supplied-Air Respirator</u>
Air-supplying respiratory protection equipment is required under the following circumstances:

o Oxygen deficient atmospheres.

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o Toxic organic vapors in excess of their IDLH concentrations, unknown organic vapors in excess of five

parts per million, or toxic organic vapors that do not have an approved air-purifying cartridge.

- o Flammable atmospheres.
- o Any other IDLH conditions.
- o In vapor concentrations greater than the protection offered by air-purifying respirators.
- o When operations are being performed that could result in a sudden and unpredictable excursion of toxic or flammable vapors or gas.

Few operations to be conducted in performance of this investigation are expected to require the use of Self-Contained Breathing Apparatus (SCBA's) or Supplied-Air Respirator (SAR's). Organic vapors will be presumed to be exclusively those of MIBK unless there is substantial evidence to the contrary. MIBK is efficiently adsorbed on activated carbon-based air-purifying respirator cartridges, has excellent warning properties, and a high IDLH level (5,000 parts per million).

The site has been occupied for approximately 30 years and hazards are well characterized for the most part. Most activities in support of this remedial assessment will not involve operations that could result in exposure to unknown chemical species or result in sudden releases of toxic vapors or gases.

SCBA or SAR will be used in the following operations or circumstances:

- o Entering confined spaces that have less than 19.5 percent oxygen as measured on an electrolytic oxygen meter.
- o Organic vapors in excess of 1,000 parts per million as measured on a field OVA utilizing either a flame or photoionization detector.
- o Organic vapors in excess of five parts per million in the absence of the characteristic odor of MIBK.
- o Entering atmospheres in excess of 25 percent of the lower flammability limit except where the atmosphere is in a confined space (in which circumstance the action level will be 10 percent of the lower flammability limit).

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o When conducting intrusive explorations in areas that may have been used for the disposal of drums or other containers of concentrated waste materials.

# B.6.2 <u>Air-Purifying Respirators</u>

Air-purifying respirators (APR's) will be used when dusty conditions are present which might result in the inhalation of radionuclides or when organic vapors at or near the PEL are present. Organic vapors will be presumed to be MIBK unless there is good reason to believe otherwise since this is the only organic chemical used in the manufacturing process. (If other organic chemicals such as cleaning solvents are used in the course of the project, the PEL for that substance will be used for setting appropriate respiratory protection.) Any visible dust in the air will result in the use of an APR for dust and radionuclide removal.

A half-face APR may be used for dust protection when provided with an MSA Type 'S' or Type 'H' filter cartridge. A half-face respirator may be used for organic vapors up to 250 parts per million presumed MIBK. Between 250 parts per million and 1,000 parts per million presumed MIBK, a full-face APR must be used because of its greater protection factor. If both organic vapor and dust are present, combination cartridges such as the MSA Type 'GMA-H' or equivalent must be used. Auxiliary dust and mist filters such as the MSA Type 'F' are not rated for control of radionuclides.

## B.6.3 <u>Mechanical Protection Devices</u>

These include items such as hard hats, safety glasses, and hard-toe boots. These items will be worn at all times when conducting field activities in support of this remedial assessment. Other protective devices such as additional foot protection, splash shields, blast protectors, etc. may be specified as needed based on site conditions and work being performed.

#### B.6.4 Protective Clothing

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The following items of protective clothing may be required depending on the type of job being performed:

- o Chemically resistant boots
- o Chemically resistant gloves
- o Plain (uncoated) Tyvek

- o Chemically resistant Tyvek
- o Fully encapsulating suit

It is not likely that any tasks will be performed in the course of this investigation that will require the use of a fully encapsulating suit. This type of garment is required only when performing tasks with a high probability of gross contamination from concentrated hazardous wastes or in highly contaminated atmospheres where the barrier properties of chemically resistant Tyveks are not sufficient. Neither circumstance is anticipated in this project. However, buried drums may be found that will necessitate an immediate removal for which use of this type of protective clothing could be required.

Chemically resistant boots will be worn whenever work activities could reasonably be expected to bring a worker into or onto hazardous chemicals or soil or other material contaminated with hazardous chemicals. Most sampling operations that will be conducted under this remedial assessment will require the use of chemically resistant boots. One of the most ubiquitous problems at this site is the presence of dust and soil with elevated concentrations of radionuclides. Leather boots or shoes can trap this material and allow it to be removed from the site and even taken home by the employees. Chemically resistant boots, because of their material of construction, are not as likely to trap and retain these particulates. They can also be decontaminated easily.

Whenever potentially contaminated materials are to be handled during the sampling operation, chemically resistant gloves will be worn. This will be the case for virtually all sampling activities. Inner gloves will be worn at the direction of the site safety officer.

Uncoated Tyvek garments will be worn to protect workers' skin and clothing from potentially hazardous dusts and soil. Whenever work is being performed in dusty conditions, uncoated Tyveks will be worn to keep this potentially contaminated material from getting on employees' clothing where it may be removed from the site and enter the general environment outside the plant facility.

Coated chemically resistant Tyveks will only be worn where activities that involve the likelihood of moderate spillage or splashing of hazardous materials

or substantially contaminated environmental materials exists or where there are high levels of organic vapors. These conditions will certainly apply when employees are involved in sampling the ponds and any containers of unknown chemicals that may need to be opened. The type of chemically resistant coating will be established by the site safety officer based on site observations and the hazardous constituents known or believed to be present. Tyveks coated with Mylar (Saranax) will be used in all operations involving sampling or handling of semiliquid or liquid process residues in or derived from Pond No. 3 or when corrosive materials from other sources must be handled or sampled. Polyethylene-coated Tyveks may be used in other situations requiring chemically resistant garments that do not require the higher level of skin protection afforded by the Saranax suit.

Other protective clothing may be required from time to time based on site conditions or tasks being performed. The site safety officer will specify any additional protective clothing that may have to be worn.

# B.6.5 Other Protective Equipment

The site safety officer in consultation with project management and the health and safety coordinator may specify other items of personal protection equipment as required to protect workers in the course of their performance of project activities. Examples of such protective equipment that particular tasks may require include the following:

- o Safety harness
- o Lifelines

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- o Extraction equipment
- o Escape air-supply devices
- o Disposable oversuits
  - o Other protective clothing items
  - o Communication devices

Employees will be trained in the method and conditions of use of such equipment before they will be required to use it.

#### B.7.0 Medical Surveillance Plan

No project-specific medical surveillance plan is proposed. Any employee who believes that he has been overexposed to a toxic chemical or who develops symptoms that could reasonably indicate an overexposure to a toxic chemical will be offered appropriate medical consultation and follow-up should the examining physician so recommend.

# B.8.0 Environmental and Personal Monitoring Plan

Environmental and personal monitoring will be carried out to determine two separate but related tasks. The first is to determine the exposure of employees to toxic substances and radioactivity so that effective personal protection measures can be taken. The second is to determine whether or not (and if so how much) contamination may be leaving the site during the course of activities taken to conduct the assessment. This HSP addresses only the environmental and personal monitoring related to employee health and safety.

# B.8.1 Monitoring Instruments and Frequency

The following instruments will be used during field operations performed in support of the remedial assessment:

- o Oxygen meter
- o Flammable atmosphere indicator
- o OVA (either FID or PID).
- o Radiation detector capable of responding to alphaparticle radiation
- o Bulk-air sampling pumps
- o Specific gas detector tubes

## B.8.1.1 Oxygen Detector

printing to the

Oxygen content in air will be determined prior to any employee entering an excavation, manhole, or other confined space. While an employee is within the confined space, his breathing air will be monitored for oxygen content on a

continuous basis. A level of 19.5 percent oxygen must be maintained at all times or else positive ventilation must be implemented or SCBA or SAR worn.

### B.8.1.2 Flammable Atmosphere Indicator

This instrument will be used whenever work is being performed around bulk amounts of MIBK or other flammable liquid and whenever total organic vapor in the air is measured at levels in excess of 100 parts per million. The flammable-atmosphere indicator will be used prior to any confined space entry operation and at any other time or place specified by the health and safety officer. Monitoring will be continuous during confined-space activities.

#### B.8.1.3 <u>OVA</u>

Either PID or FID devices will be acceptable for this job. If a PID device is used, a lamp energy of 11.7 eV is recommended since this gives a response factor of 1.06 (i.e., essentially a direct-reading true value). If a 10.2 eV lamp is used, the response factor is only 0.57. These instruments will be used whenever work is being performed in or around Pond No. 3, materials derived from process wastes containing MIBK are being sampled or handled, unknown materials are encountered, and whenever a new work area is entered or a new task commences. During excavations, subsurface sampling, and monitoring well drilling, organic vapor readings will be taken on a frequent basis as established by the site safety officer based on his observations at the time.

# B.8.1.4 Radiation Detector

A radiation detector will be used for all sampling activities and to make measurements of ambient radiation levels. Radiation measurements will be made at each work site. Samples will also be scanned for radiation prior to packaging for shipment to the laboratory.

#### B.8.1.5 Bulk-Air Sampling Pumps

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From time to time during the remedial assessment, the site safety officer will take samples of breathing-zone air to determine the concentration and identity of airborne contaminants. These samples may measure fluorides, acid gases, organics, or radiation. Flow rates and sampling media will be as specified in the most-recently published NIOSH methods manual or other approved method absent a NIOSH method for the material in question.

#### B.8.1.6 Detector Tubes

Specific compound detector tubes may be used whenever instrument readings indicate that organic vapors are present or when the presence of acid gas vapors is suspected. The occasions and frequency of measurement with detector tubes will be determined by the site health and safety officer.

## B.8.2 Maintenance and Calibration

All field direct-reading monitoring instruments except the radiation detector will be calibrated daily against test gases of known concentration. The radiation detector will be calibrated by the vendor at such frequency as he deems appropriate. Calibrations will be performed in accordance with the manufacturer's recommendations. A written record of the calibration will be made (or maintained on the site in the case of the radiation detector) each time the calibration is performed.

Maintenance of monitoring instruments will be performed as needed. Instruments will be inspected for proper functioning each time calibration is performed. Any maintenance needs identified during the inspection will be promptly attended. If any components have a specifically identified useful lifetime, those parts such as oxygen detector cells will be replaced as needed.

#### B.8.3 <u>Personal Dosimeters</u>

All Earth Sciences personnel who will perform site operations that will or could result in exposure to elevated levels of radioactivity will be enrolled in a dosimeter program. Thermoluminescent dosimeters will be used since they will respond to alpha-particle exposure. Dosimeters will be read on a monthly basis. Employee exposure records will be maintained for all employees in the program.

## B.9.0 Site Control Plan

Site control plans will be implemented in all areas of the facility that will be subject to any sort of intrusive activity. Work areas will be flagged in the field and also indicated on site maps. Work areas so flagged will constitute exclusion zones and appropriate work practices will be used in all such areas. These work practices include the following:

- o Establishment and use of controlled access points
- o Use of required PPE
- o Mandatory use of buddy system
- o Logging all personnel in and out
- o Establishment of decontamination facilities
- o Agreement on communication signals

Establishment of control-zone boundaries will be performed by the site safety officer in coordination with the site supervisor or project manager.

#### B.10.0 Decontamination

All personnel and equipment that leave an exclusion zone will have to be properly decontaminated. Detergent and water will be adequate to remove the materials known to be present including the radionuclides.

The details of each decontamination facility's layout and number of separate cleaning stations will be established by the site safety officer based on the anticipated degree of contamination present and the tasks being performed. He will also be responsible for prescribing additional decontamination products if any should be found to be necessary.

Decontamination residues will be stored in appropriate containers until suitable arrangements can be made for their disposal. Residues will be tested for both chemical and radioactive contamination before any decision is made concerning mode of disposal.

Periodic wipe samples will be obtained from areas outside the decontamination facility. These wipes will be tested for fluorides, pH, and radioactivity to determine the effectiveness of the decontamination procedures used.

If equipment or tools must be decontaminated at some location other than the designated decontamination facility, they should be carefully wrapped in a plastic bag and the bag sealed appropriately. Any decontamination residues must be properly managed.

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### B.11.0 Confined Space Entry

All confined-space entry activities will be conducted in accordance with the Earth Sciences Confined-Space Entry Procedures. A copy of this procedure is included as Appendix A of this health and safety plan.

## B.12.0 Spill Control Plan

There are relatively few situations in the assessment that will require implementation of a spill control or containment plan since most activities will be restricted to obtaining samples of various materials on the site. However, the following spill potentials do exist and must be addressed:

- o Fuel spills or leaks from drilling equipment
- o Rupture or other failure of containers being sampled
- o Embankment failure
- o Loss of sample

Fuel spills will be managed by a regular program of service and maintenance of drilling equipment to insure that they are properly maintained. If equipment is to be refuelled on the premises, this will be done in a designated area where spill control equipment or facilities are available. Spill control equipment for equipment refueling will consist of a supply of suitable absorbent material.

It may be necessary to sample material in drums or other containers. If this activity must be performed, no container will be opened unless and until there is adequate spill control equipment and support. Spill control equipment and supplies will consist of the following:

- o Overpack drums
- o Absorbent pigs
- o Granular absorbent material
- o Salvage drums
- o Nonsparking tools
- o Transfer pumps

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One of the materials that may be present in drums or other portable containers is hydrofluoric acid. All materials that may be used in spill control must be

resistant to HF if there is any likelihood that this corrosive material is present.

An embankment failure at any of the impoundments could result in a major release of material. Work with heavy equipment or conducting borings or excavations on or near the embankments could cause a failure or encounter a piping zone. If this should occur, earth-moving equipment will be used to erect temporary dams or embankment to prevent the material from reaching the river. Escaped material will then be managed as appropriate to its chemical composition.

Loss of sample will result in only localized spillage. Lost sample materials will be cleaned up using absorbent material or by shoveling the sample and any affected soil into a suitable waste container.

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Appendix C

Standard Earth Sciences Consultants, Inc.'s Forms



Project No	Project Name _		Sheet	of
Field Activity		Location	Date	·
Daily Activities (contir	nue on reverse side if n	ecessary):		<del></del>
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<del></del>	!	Visite as On Otto		<del></del>
ather Conditions:		Visitors On Site:		
th Sciences Personn				

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					Boring Log		
Client					Project No E	Boring No	
I	on					ield Scientist	
l						Checked By/Date	
						Page o	
Dato	1						•
Depth (Feet)	Sample No. and Type	SPT Blows (6") or RQD (%)	Sample Recovery	Profile	Profile Description (Includes Color and Density/Hardness	s)	Well/Piezometer Construction Detail
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			Boundary				· · · · · · · · · · · · · · · · · · ·			
	ıntist	Date	Coarse Fragments (% and description)							
	Field Scientist		Moist Consistence							
Description		Soil Series	Structure					·		
Soil Profile Description		r	Mottles (description and color)	,			·			
	ате		Texture							
	Project Name	Location	Matrix Color						·	
	Project No.	Test Pit No.	Depth (inches)	1	1					al Notes:
	يّ		Horizon							Additional Notes:



5	Slug Test Data Sheet
Project Name	Well Identification
Project No User	Hermit Identification No.
Well Diameter	Start Date/Time
∇ Before Slug Introduction	
√     After Slug Introduction	Type of Transducer (PSI)
After Slug Removal	Depth of Transducer
	TOC Elevation
Type of Test (check one):	Rate (check one):
Rising Head	Logarithmic
Falling Head	Linear
Reference Level Set Yes/No	Display Mode (check one):
	English (Surface)
Scale Factor	English (TOC)
	SI (Surface)
Offset	SI (TOC)
Select File No.	Diameter of Slug (i.e., 1-1/2", etc.)
	Length of Slug (volume, if known)
Weather Conditions:  Note: Do not terminate test until water level has	
Comments:	—
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Project Name	Sample No.
Project No.	Groundwater Level Elevation (ft/msl)
W	/ell Evacuation Information
Upgradient Downgradient	Measurement Reference Point:
Well No.:	Casing Stickup (ft):
Evacuation Date:	Total Well Depth (D) (ft):
Evacuation Time:	Initial Water Level (I) (ft):
Evacuated By:	Radius of well (r):
Evacuation Method:	Gallons per foot: $(G = \pi^2 \times 7.48)$ :
Time pumping began:	Well Volume (gal): G x (D-I):
Time pumping ended:	Total Gallons Removed:
Total pumping time:	Well Volumes Removed:
Pumping rate:	
	Sampling Information
Sample Date:	Water Level Before
Time Collected:	Sampling:
Collected By:	Depth Sampled:
Location Description:	Sample Types:
Well Pond Seep	
Stream Spring	Field Measurements (make replicate measurements
Other	of pH and specific conductance):
Sample Method:	Temperature (° C):
PVC Bailer VOA Sampler	pH: Specific Conductance:
Teflon Bailer Bladder Pump	•
Other	pH:Specific Conductance:
	pH:Specific Conductance:
Field Observations:	Calibration: pH Specific Conductance:
Turbidity Odor	•
Immisible Layers	
Color	· · · · · · · · · · · · · · · · · · ·
	General Information
	General information
weather conditions.	

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						Chain of Custody Record	ody R	ecor	70								
Project No.		Project Name	ате				, to										
Samplers (signature)							00 -	<u> </u>			\					Remarks	ks
Station No.	Date	Time	7.	Comp	<del>2</del> 8	Station Location	tainers						//	/	,		
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Relinquished by (signature)	signature)		Date/Time	Time		Received by (signature)	gnature)			Ι							
Relinquished by (signature)	signature)		Date/Time	Time		Received by (signature)	gnature)			Γ							
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Relinquished by (signature)	signature)		Date/Time	Time		Received for Laboratory by (signature)	boratory b	y (signa	ature)	Ī							
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	Bottle/Analytica	l Work Request - Liquids	;
Project No.	Project Manager	Work requested by	
			_
Invoice to	Hesuits to	Table of results format	
		<u> </u>	special (attach example)
	time am pm	Gen. Chemical Analyses	Metals Analyses 1
Date of bottle order:		Acidity	Aluminum (Al) F H
Date of bottle order.		Alkalinity	Antimony (Sb) F H
Date bottles needed:	1 1 1	Amonia	Arsenic (As) F H
Bata kamba assaliwadi		BOD (5 day)	Barium (Ba) F H
Date bottles received:		Bicarbonate	Beryllium (Be) F H
Date results needed:		Bromide	Boron (B) F H
<u> </u>		COD	Cadmium (Cd) F H
Bottle T	vines	Carbon (TOC)	Calcium (Ca) F H
to be filled out	אָרָכּיּ t by Antech)	Carbonate	Chromium (Cr) F H
Chemistry	<del></del>	Chloride	Chromium (Cr+6) F H
(no preservative)	Metals (HNO3)	Color	Cobalt (Co) F H
1 liter (p)	500 ml (p)	Cyanide (free)	Copper (Cu) F H
500 ml (p)	250 ml (p)	Cyanide (total)	Iron (Fe) F H
250 ml (p)	125 ml (p)	Cyanide (amendable)	Iron (Fe+2) F H
125 ml (p)	special	Fluoride	Lead (Pb) F H
		Halogens	Magnesium (Mg) F H
Nutrient (H2SO4)	Organic	Hardness (Ca = Mg)	Manganese (Mn) F H
	(no preservative)	Hydroxide Nitrate	Mercury (Hg) F H
500 mt (p)	2-1/2 liter (g)	Nitrogen-kjedahl	Molybdenum (Mo) F H
250 ml (p)	1 liter (q)	Oil & Grease	Nickel (Ni) F H Potassium (K) F H
125 ml (p)	40 ml VOA	Osmolality	
TOV (Nesco)	TOC	DH OSTROIAINY	Selenium (Se) F H
TOX (NaSO3)	(no preservative)	Phenolics	Silicon (Si) F H
500 ml (g)	40 ml VOA	Phosphorus (Ortho, Total)	
250 ml (g)	120 ml bottle (q)	Specific Conductance	
		Sulfate	Thallium (Ti) F H
Cyanide (NaOH)	;Oil & grease (HCI)	Sulfide	Titanium (Ti) F H
1 liter (p)	1 liter (g)	Sulfite	Vanadium (V) F H
500 ml (p)	Titlet (d)	Surfactants (MBAS)	Zinc (Zn) F H
7 300 (III (D)	Others	Turbidity	(2110)
Phenolic (H2SO4)		Residue Solids	Analytical List
1500 11/2		Filterable (TDS)	Priority Pollutants
500 ml (q)	<u> </u>	Nonfilterable (TSS)	Target Compounds
Additional Instructions:		Settleable (Imhoff)	Ohio Appendix A
	•	Total	Form 8
1			Form 19 Quarterly
		Organic Analyses	Form 19 Annual
	r	Pesticide	
		Herbicide	Other Analyses
		Polychlorinated biphenyls (PCBs) water	<del></del>
1		(PCBs)     Water	
	a .		
·	· * · · ·	Volatiles Acid Futrostobles	
1		Acid Extractables	
		Base-neutral Extractables	

<sup>1</sup> F= flame; H = furnace Rev. 5/31/90



1	Bottle/Analytical W	ork	Request - Solids/Semi-So	illds
Project No.	Project Manager		Work requested by	
Invoice to	Results to		Table of results format	standard
				special (attach example)
	time am	pm	Gen. Chemical Analyses	Metals 1
Date of bottle order:	unie an	ا تــــــــــــــــــــــــــــــــــــ	Acidity	Aluminum (Al) F H
Date of bottle order.		$\perp \downarrow \downarrow \downarrow$	Alkalinity	Antimony (Sb) F H
Date bottles needed:	1 1		Ammonia	Arsenic (As) F H
Date bottles received:		$\Box$	Bromide	Barium (Ba) F H
		╂╼╣	COD	Beryllium (Be) F H
Date results needed: · ·		$\perp \downarrow \mid$	Carbon (TOC)	Boron (B) F H
		===	Carbonate	Cadmium (Cd) F H
	Types		Chloride	Calcium (Ca) F H
}	ut by Antech)	<b>-,</b>	Cyanide (free)	Chromium (Cr) F H
Soil	Others	_] [	Cyanide (total)	Chromium (Cr+6) F H
(no preservative)		]	Cyanide (amendable)	Cobalt (Co) F H
1,000 ml		_]	Fluoride	Copper (Cu) F H
250 ml		┙╽	Halogens (TOX) Hardness (Ca + Mg)	Iron (Fe)
30 ml	1	i	Nitrate	
	• 	]	Nitrogen-Kjedahl	Magnesium (Mg) F H
Additional Instruction	s		Oil and grease	Manganese (Mn) F H
II .		- 1	HqH	Mercury (Hg) F H
11		- 1	Phenolics	Molybdenum (Mo) F H
]]		1	Phosphorus (ortho, total)	Nickel (Ni) F H
11		- 1	Specific conductance	Potassium (k) F H
{{ ·		Į	Sulfate	Selenium (Se) F H
			Surfactants (MBAS)	Silicon (Si) F H
<b>}</b> }		1	Miscellaneous Analyses	Silver (Ag) F H
11		- 1	Neutralization	Sodium (Na) F H
11			Potential acidity	Thallium (TI) F H
11		l	Leachate generation	Tin (Sn) F H
11		1	ASTM	Titanium (ti) F H
		1	EP toxicity	Vanadium (v) F H
11		- 1	Texture	<u> </u>
]] .		1	pH Specific conductance	Organic Analyses
11		· \	Organic matter	Pesticide Herbicide
<b>{</b> {			Sulfur species	Polychlorinated biphenyls
[]			Total	(PCB's) water
]]		Ì	Pyritic	□ oil □ solid
<b>}</b>	4		Sulfate	Volatiles
11		ļ	Organic	Acid extractables
11		İ	SAR	Base-neutral extractables
11	1	Ì	CEC	Analytical List
11		}	Weak acid extractable	Priority Pollutants
11		į	Anion exchange capacity	Target Compounds
11		I	Total kjeldahl Nitrate	Ohio Appendix A
11		j	Phosphate	Form 8
<b>{}</b>		1	Chloride (soluble)	Form 19 Quarterly
		ļ	Sulfate (soluble)	Form 19 Annual
		ļ	Residue Solids	Other Analyses
			Filterable (TDS)	
]]		ĺ	Nonfilterable (TSS)	
			Total	
L				

1 (F= flame; H= furnace) Rev. 5/31/90



	Bottle/Analytical V	/ork	F	Request - Solids/Semi-S	oli	ds	
Project No	Project Manager			Work requested by			
Invoice to				Table of results format			
						special (attach example)	
	time ar	n pm		Gen. Chemical Analyses	]	Metals 1	
Date of bottle order:				Acidity	1		Щ
Date bottles needed:		77		Alkalinity	1		버
	<del> </del>	-+-		Ammonia	┨		出
Date bottles received:		44		Bromide COD	1		버
Date results needed:		] ]]		Carbon (TOC)	1		
\			!	Carbonate	]		HП
	Types			Chloride	1	Calcium (Ca) F	н
1	out by Antech)	,		Cyanide (free)	1	Chromium (Cr) F	
Soil	Others		}	Cyanide (total)	4		ഥ
(no preservative)				Cyanide (amendable)	┨		븼
1,000 ml	1		Į.	Fluoride	-		버
250 ml	1 L	]	1	Halogens (TOX) Hardness (Ca + Mg)	1		붜
30 ml	1	1		Nitrate	┨		H.
				Nitrogen-Kjedahl	1		出
Additional Instruction				Oil and grease	1		щ
<b>{                                    </b>			l	HqH	1		H
11			l	Phenolics	1		н
				Phosphorus (ortho, total)	]	Nickel (Ni) F	H
{  .				Specific conductance	1	Potassium (k) F	
				Sulfate	4	Selenium (Se) F	Щ
				Surfactants (MBAS)	Ī		ᆈ
<u> </u>  -				Miscellaneous Analyses	]		버
11			ĺ	Neutralization	1	<del></del>	井니
{			1	Potential acidity	4		<u>Н</u> Н
	ı			Leachate generation	4		H
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	ı		1	Texture	┨		Н
				PH	1	Organic Analyses	
				Specific conductance	1	Pesticide	
<b>}</b> }-			ļ.,	Organic matter	1	Herbicide	
	. '		ļ.,	Sulfur species	]	Polychlorinated biphenyls	
[]			[	Total	]	(PCB's) water	
[]	,		ľ	Pyritic	1	oil solid	
[[			ľ	Sulfate	4	Volatiles	
			I.	Organic SAR	4	Acid extractables  Base-neutral extractables	
]]			ŀ	CEC	┨		
H				Weak acid extractable	┨	Analytical List	
			ľ	Anion exchange capacity	1	Priority Pollutants	
11				Total kjeldahl	1	Target Compounds Ohio Appendix A	
<b>{ }</b>	A programme and the second sec			Nitrate	1	Form 8	
			0	Phosphate	]	Form 19 Quarterly	
				Chloride (soluble)	]	Form 19 Annual	
	•			Suffate (soluble)	_	4 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
			ľ	Residue Solids	]	Other Analyses	
	;			Filterable (TDS)	]		
			ŀ	Nonfilterable (TSS)	1		
				Total	_		

<sup>1 (</sup>F= flame; H= furnace) Rev. 5/31/90